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# The Use of Synthetic Red Gypsum as a Construction Material

By

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A thesis submitted to the University of Newcastle upon Tyne in partial fulfilment of the requirement for the degree of Doctor of Philosophy

**Declaration**

This is to certify that I am responsible for the work submitted in this thesis, that the original work is my own except as specified in acknowledgements or within the text, and that neither the thesis nor the original work contained therein has been submitted to this or any other institution for a higher degree.

Signed .

Date.....

**Acknowledgements**

I would like to express my gratitude and sincere thanks for all the help and assistance given to me during this project, by the many people who offered it.

Firstly, I would like to thank Dr Stephanie Glendinning for her fantastic supervision, guidance, support and patience during this research programme, particularly in the final year of the project when she made it possible to juggle my time between the write up and the Bionics research programme.

Many technicians helped in conducting laboratory testing for this project, many thanks go to Chris Hunt, Fred Beadle and Stuart Patterson in the soil mechanics laboratory; Billy Cragie in the concrete laboratory; Kath Liddell in the XRD laboratory; and Grant Staines in the SEM laboratory. Members of the soil science research group are also acknowledged for their help in advising on mineralogical testing. I would also like to offer sincere thanks to Denis Kalumba and Addy Kho for their assistance with the concrete testing.

The project was funded by the Engineering and Physical Science Research Council (EPSRC) with additional funding provided by Huntsman Tioxide Europe Ltd. Thanks go to Brian Noble and Jean Coulon of Huntsman for their advice and support throughout the project. The two other industrial partners, Hercules Stent and Faber Maunsell are also acknowledged for their contributions to the project.

I would also like to thank Nick, Hannah, Kate, and Dawn plus all my other friends and colleagues who have offered help and understanding through the difficult periods that almost all research projects produce.

Finally, I am eternally grateful to my parents for their support and encouragement throughout the 4 years of this project.

**Abstract**

Huntsman Tioxide produce a co-product "red gypsum" (red due to iron content) as a filter cake during the neutralisation of sulphuric acids at the end of the Titanium Oxide production process. Globally, Huntsman produce 925 000 tonnes per year of red gypsum. The majority of the material goes to landfill, the rising cost of which has made it essential to find alternative uses.

At present cementitious binders are used extensively in the construction industry, principally in concretes but also in applications like ground improvement. In these applications the cost of the binder, typically Portland cement, makes up a considerable percentage of the overall cost of the technique. In addition to the financial cost there is also the environmental cost of quarrying and processing of materials to produce Portland cements. Gypsum based industrial bi-products have been identified as alternative sources of cement (Beretka et al, 1996). Using these materials has two advantages: they have little or no production cost; and the re-use of such material would negate the need for expensive disposal.

This thesis describes a programme of laboratory testing and field trials to investigate the potential of using synthetic red gypsum as a construction material. The main applications investigated were deep dry mix soil improvement and the production of paving blocks.

Laboratory trials investigated the properties of red gypsum on its own and when mixed with Pulverised Fuel Ash, Ground Granulated Blast Furnace Slag, Lime and steel slag at a range of water contents. An assessment of samples was made on the basis of Unconfined Compressive Strength at 28 days curing. It was found that a red gypsum : Ground Granulated Blast Furnace Slag mix achieved the highest unconfined compressive strengths (up to 39 MPa) and was selected for further investigation as a binder.

This binder was then mixed with a range of soils in the laboratory, it was found that red gypsum based binders can perform as well as Portland cement as a soil mixing binder, and that concrete blocks can be produced with strengths approaching that of equivalent Portland cement mixes. A field trial was also conducted in which red gypsum binders to investigate whether the binder would work in situ. It was found that the red gypsum binder performed adequately to pass standard engineering specifications for soil mixing.

The thesis concludes that there are several potential applications for the use of red gypsum in the construction industry but that further work is required before it can be used commercially.

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# Chapter 1

# Introduction

## 1. Introduction

This thesis investigates the potential for the use of co-product Red Gypsum (from the production of titanium dioxide pigment) as a construction material. The focus of the research concentrates on developing and maximising the pozzolanic reactions within the Red Gypsum by mixing it with a range of waste materials to create a binder. Research then concentrates on developing the use of this binder.

This research has been motivated by the economic and environmental concerns over the disposal of wastes coupled with the costs of traditional engineering materials. The use of "waste" materials has already become fairly commonplace, a considerable market being formed for materials like Ground Granulated Blast Furnace Slag (GGBS) and Pulverised Fuel Ash (PFA). Their use is now accepted in construction engineering.

Titanium dioxide pigment produced by the sulphate process results in the formation of spent sulphuric acid, which is neutralised by the addition of limestone. This yields two gypsum co-products, referred to as red and white gypsum. Applications for co-product white gypsum are already well established, white gypsum being a principal ingredient in the production of plasterboard. Currently co-product red gypsum is being utilised only in agriculture as a soil conditioner.

Studies have been carried out recently to investigate other uses of co-product red gypsum within the construction industry (Grant 1997, Anon 2001 and Simpson 2001). These are reviewed in Chapter 2, and identify the potential for co-product red gypsum to be used as a soil mixing agent, particularly when mixed with GGBS.

For ease of reference throughout this thesis co-product red gypsum will be referred to simply as Red Gypsum.

### 1.2 Aims and Objectives of this Study

The previous studies conducted on potential engineering uses of Red Gypsum have indicated that when mixed with GGBS the optimum ratio for achieving high strengths

is 2:1 GGBS to Red Gypsum (Anon, 2001 and Simpson 2001). The aim of this research is to investigate the potential of mixing Red Gypsum with various "wastes" (including GGBS) at various ratios at a range of different water contents to maximise strength gain. The research then aims to test whether the optimised material is suitable to be used as an admixture for the engineering improvement of a range of soils and as a replacement for Portland cement in the production of concrete products. The final aim is to understand which minerals formed during the hydration of the Red Gypsum mix are responsible for the increase in strength.

In order achieve these aims a series of laboratory test programmes have been conducted. The specific objectives of these programmes are to:

- Develop a binder (with advantageous strength properties) by mixing Red Gypsum with GGBS, PFA, and Steel Slag at a range of water contents. In order to achieve this, a series of tasks must be undertaken:
    - Establish which waste has most potential for developing the strength of Red Gypsum.
    - Identify what water content should be used to maximise the strength gain.
    - Determine the effect of density on the binder strength.
    - Determine what minerals are being formed within the binder and are causing the strength gain using X-ray diffraction (XRD and Scanning Electron Microscope techniques).

- Assess the suitability of the developed binder as a soil mixing agent.
  Tasks:
    - Mix the binder with a range of soils, test the mixed soil at a range of curing periods to quantify changes to shear strength, stiffness, strain at failure, density, and water content over time.
    - Test the binder in both laboratory and in-situ conditions.
    - Determine which soils have the greatest potential to be improved by Red Gypsum binders.

- Compare the effectiveness (with respect to strength and stiffness) of Red Gypsum binders with Portland cement in soil mixing applications.
- Determine what minerals are being formed within the binder-soil and are causing the strength gain using X-ray diffraction (XRD and Scanning Electron Microscope techniques).
- Assess the freeze-thaw and wet-dry durability of soil improved using Red Gypsum binder.

- Assess the suitability of Red Gypsum binders as replacements for Portland cement in the production of concrete blocks.

  Tasks:
  - Determine the effect of binder composition on the slump workability of fresh mortar and concrete mixes, compare this to Portland cement.
  - Determine the effect of binder composition on the strength of mortar and concrete samples over time.
  - Assess the effect of initial mix water content on the strengths of mortars and concretes made with Red Gypsum over time.
  - Evaluate the potential of using other waste materials as replacements for sand and colouring agents within concretes made with Red Gypsum binder and Portland cement.
  - Assess the effect on colour of the use of Red Gypsum binders has on concretes.
  - Determine what minerals are being formed within the Red Gypsum concretes using X-ray diffraction (XRD and Scanning Electron Microscope techniques).

### 1.3 Overview of Project

A review of previous research into the use of waste materials in civil engineering, including recent work carried out on Red Gypsum, is presented in Chapter 2. In addition common cement and pozzolan reactions are described along with the origin and formation of the various waste materials used in this study. The relevant legislation is also briefly reviewed.

With the exception of the field testing which is covered in Chapter 7, the testing methods and programmes which make up this study are explained in Chapter 3. Descriptions and geotechnical index properties (Atterberg limits etc) are also presented in this chapter.

Chapter 4 presents the results of the binder development work; Chapter 5 the laboratory results from the soil mixing study. Chapter 6 shows the results of the concrete testing, and Chapter 7 the results of a field trial of in situ soil mixing. At the end of each of these chapters the results of each individual test programme is discussed. Combined discussion and conclusions of the whole study are presented in Chapter 8, together with recommendations for further study.

# Chapter 2

# Literature Review

# 2. Literature Review

## 2.1 Introduction

The introduction of landfill tax and stricter control of planning consents for new disposal and tipping sites in the UK has been an effective method of stimulating efforts to find alternate uses for many wastes.

The public perception of re-cycling is predominantly focussed on domestic wastes, (paper, glass, plastics) but these make up only a small proportion of waste produced in the UK. Fossil fuel based energy production, the steel industry, the chemical industry, the construction industry and mining produce millions of tonnes of waste each year, most of which is tipped in spoil mounds or buried in landfill. Unlike materials like glass, for example, these materials are not regarded as having any value, so until the recent introduction of legislation there has been little effort to find alternate uses. The construction industry uses massive volumes of bulk material each year as fill and in concretes and it is seen as an ideal industry to utilise "wastes".

In this chapter lime is referred to frequently, where the term unslaked lime is referred to this means calcium oxide (CaO), where slaked lime is referred to this means Calcium Hydroxide ($Ca(OH)_2$ .

## 2.2 Previous Use of Wastes and Co-Products in Construction

Some utilisation of bulk industry wastes within the construction industry has already taken place and research is being conducted into finding uses/innovative disposal options for others. Three of the main areas where research has been concentrated are

- Cements and concretes
- Waste aggregates
- Ground improvement
- Ground remediation

### 2.2.1 Wastes in Cements and Concretes

Most concretes are prepared with some mineral additions in their composition. The different types of additions include natural pozzolans, fly ash, silica fume, blast furnace slag. Calcareous fillers are also often included. Structural concretes have been designed to include very high volumes of fly ash or blast furnace slag (Giaccio and Malhotra, 1988). In this sense, Portland cement materials appear as an alternative for finding ecological solutions to safe disposal of waste materials, (Schissl and Hohberg, 1997). Pulverised Fuel Ash (PFA) has been added to cements in small quantities to achieve high strength concretes and Ground Granulated Blastfurnace Slag (GGBS) used to make sulphate resistant cements, and as an additive to Ordinary Portland Cement (OPC) for the stabilisation of organic soils.

#### 2.2.1.1 Waste Gypsum

Beretka et al (1996) identified calcium sulphoaluminate based cements as worthy of consideration, these cements can be made by firing industrial wastes and by-products at relatively low temperatures (1200°C). Calcium sulphoaluminate based cements show rapid hardening due to the rapid formation of non-expansive ettringite ($C_6AS_3H_{32}$) which originates from the hydration reaction of calcium sulphoaluminate ($C_4A_3S$) and calcium sulphate. Ettringite develops relatively large crystals able to produce high mechanical strengths at early stages in the curing process, (Beretka et al,1996). CSA cements are used in applications where high early strength, impermeability, and sulphate resistance are important. These cements should not be confused with super sulphate cement which does not develop high strengths.

The main sources of chemical gypsum are the manufacture of mineral acids by attack of natural rocks with sulphuric acid, then the neutralisation of acid waste with lime.

Different types of chemical process produce different types of calcium sulphate, di-hydrate ($CaSO_42H_2O$), hemihydrate or anhydrite ($CaSO_4 \cdot 0.5H_2O$). Calcium sulphate di-hydrate, the most frequent phase of chemical gypsum, is mainly used as a set retarder in the cement industry and as a raw material for the manufacture of plaster products and preformed building elements. Beretka et al (1996) commented that some impurities are usually present in chemical gypsum and the effects of these impurities can considerably affect the physical and visual characteristics of the commercial products. In some cases modification of the production processes may be necessary in order to avoid unfavourable effects. Chemical gypsum can also be used in agriculture for the treatment of alkaline soils, (Miller et al, 1986; Pavan and Bingham ,1986), the reactions utilized in this improvement are cation exchange, the same reactions which are used in soil stabilisation.

Beretka (1996) concluded that, given the massive amounts of chemical gypsum produced annually, the material is under utilised as a source material for calcium sulphoaluminate cement. Beretka also concluded that other wastes and by-products like blastfurnace slags and fly ash could be utilised in the manufacture of calcium sulphoaluminate cements. However, Beretka failed to note the potential of using gypsum wastes as pozzolanic cements, especially when used in combination with fly ash and blast furnace slags. However, whilst producing calcium sulphoaluminate cement from waste gypsum requires relatively low temperatures (1200°C) when compared to conventional cement production, the energy cost of this process is still high when compared to the drying and grinding of waste gypsum and mixing with slags or ashes. This should be borne in mind when considering the whole life of environmental impact of this form of waste re-use.

#### 2.2.1.2 Ashes and Slags

**Power Stations**

Ashes and slags are produced in bulk by coal fired power stations, the incineration of domestic wastes, and steel and iron production; only a limited amount is re-used. Ashes are being considered for use in cements. Jaturapitakkul and Cheerarot (2003) investigated the potential of using bottom ash from coal fired power stations as a

pozzolanic replacement for Type I Portland cement. Ghafoori and Bucholc (1996) and Bucholc (1997) had previously investigated its potential for use as a fine aggregate in cements, and Churchill and Amirkhanian (1999) investigated its use as a fine aggregate in asphaltic concrete. They concluded that the large particle size and high porous surface resulted in higher water requirement and lower compressive strength. Good results have been obtained when bottom ash is used as a fine aggregate in roller compacted concrete (Ghafoori and Cai 1998 a and b).

Juturapitakkul and Cheerarot (2003) replaced Portland cement in 10, 20 and 30% amounts with bottom ash and ground bottom ash by dry weight in mortar and concrete mixes. Specimens were mixed and cast in accordance with American Standards for Testing Materials (ASTM) part C 109. Bottom ash samples were compared with samples made with standard OPC. Scanning Electron Microscope (SEM) analysis was conducted to assess particle size and shape differences between materials, slump tests to assess workability and Unconfined Compressive Strength (UCS) tests to assess strength. Juturapitakkul and Cheerarot (2003) found that samples prepared with percentage replacements of original untreated bottom ash produced mortar specimens with considerably lower compressive strengths and higher water requirements than OPC samples. Samples prepared with ground bottom ash produced mortar samples with similar strengths and water requirements to cement samples. Juturapitakkul and Cheerarot (2003) concluded that bottom ash had a high potential for use as a pozzolanic material but that more research was required. It is unfortunate that although SEM analysis was used to assess particle size and shape of the bottom ash, no SEM analysis was conducted on the mortars and concretes produced, as this would have produced valuable evidence concerning the nature of the minerals produced during hydration.

**Municipal Solid Wastes**

Aubert, Husson and Vaquier (2003), and Goh, Show, and Cheong (2003) investigated the use of incinerated municipal waste in concretes. Goh et al (2003) conducted tests on Municipal Fly Ash (MFA) produced by incinerators equipped with lime based flue gas cleaning scrubbers. The MFA produced was used as a cement replacement at 5, 10, and 20% quantities. Samples were prepared to ASTM C109 (ASI, 2001) for compressive strength tests, and leaching tests were also

conducted. These were compared with control samples made with OPC. Goh et al (2003) concluded that samples made with the MFA-OPC blended cement had higher rates of strength development than the control samples and that equal or better compressive strengths were attained for samples tested up to 180 days curing, where cement replacement was 10%. However, considerable strength reduction occurred at replacement levels beyond 15%. Leaching tests concluded that the blended cements would not pose any long term environmental problems such as land and groundwater contamination.

Aubert et al (2003) tested the properties of mortars and concretes produced with partial cement replacement with MFA produced by the Revasol process. This process had been developed by Solvay, (a waste incineration company) and the Universite Libre de Bruxelles. The process consists of three successive steps:

- Water dissolution of the ash to remove salts
- Phosphation with phosphoric acid to stabilise metals
- Calcination to eliminate organic compounds

Mortar samples were prepared with 12.5% and 50% replacement of Portland cement with the MFA. Control samples were made with 12.5% and 50% of the Portland cement having been replaced with sand. Compressive strengths of the samples were measured in accordance with NF P18-406 (NF, 1981). Leaching tests were carried out on samples in accordance with NF PX31.210 (NF, 1992) and NF PX31.211 (NF, 1994). Incorporation of MFA in the mortar led to a slight drop in the workability, and a similar reduction was also noted in the sand replacement samples. The compressive strength of the samples with 12.5% and 50% cement replacement with MFA were lower than samples prepared with 100% Portland cement but very similar to samples where sand had replaced cement. The 50% cement replacement had lower compressive strength than the 12.5% samples. Aubert et al (2003) concluded that the substitution of MFA in place of cement in concrete did not cause loss of mechanical strength greater than that caused by the reduction in the quantity of cement, and that the MFA behaved like an inert sand. Leaching tests carried out on the concrete showed that the materials produced had

lower pollutant potential than MSWI bottom ashes which are accepted for use in roads. The use of treated ashes in hydraulic concrete is potentially profitable and provides an interesting alternative to surface dumping.

Neither Goh et al (2003) or Aubert et al (2003) conducted mineralogical analysis on the mortar and cement samples they made, so it was not possible to determine if the mineralogy of the ash cement was any different to conventional cements. Also it should be noted that in each of these studies the MFA did not produce higher performance concrete (in the case of Aubert et al (2003) it was lower) and material was effectively used as a bulk filler. Also, in each case the material used had to undergo further processing before it could be added (in the case of Aubert et al a moderately energy intensive process).

Berg and Neal (1998) analysed the possible use of municipal solid waste for the creation of concrete masonry. Ali and Chang (1994) studied the strength and durability of bricks containing ash from the incineration of municipal solid waste. Hammernik and Frantz (1991 a and b) analysed the characteristics of different municipal solid wastes and the properties of concretes with replacement of up to 60% of cement by ashes. Detrimental effects have been reported in accordance with the observations of Lavat and Tezza (1998) on the influence of lead in the inhibition of early cement hydration.

The influence of chromium, nickel and zinc on the structure and reactivity of clinker has was analysed by Stephan et al (1999 a, b and c). It was observed that these heavy metals had only minor effects on setting and hydration of cement mortars even at concentrations 10-20 times higher than those usually present in Portland cement. At very high concentrations of chromium, the rate of setting and hydration reactions increases and strength decreases. On the contrary, high contents of zinc delay setting and hydration and increase the strength, No significant effects were observed even incorporating high contents of nickel. Long term leaching studies on municipal solid waste were performed by Andac and Glasser (1999) and Hillier et al (1999) showing that toxic metals such as antimony, arsenic, cadmium, chromium, mercury, nickel, lead and selenium were not detected probably due either to a very low concentration into the cementitious matrix, or to the formation

of insoluble compounds that retain these metals. The environmental factors related to the waste disposal in concrete have also been discussed by Klitch et al (1999).

**Hospital Wastes**

Genazzani et al (2003) conducted tests using ash from the incineration of hospital waste. The ash was used as a replacement for a Portland cement in 10, 25, and 50% amounts in the preparation of mortars. A reference mortar was also mixed without ash. Samples were tested for flexural and compressive strength at 28 and 110 days. Testing showed that flexural compressive strengths reduced (by up to 50%) with increasing quantities of ash at both 28 and 110 days. Genozzini et al (2003) concluded that replacement of Portland cement with hospital waste ashes was a viable method of disposal, but further testing is required to ensure that cements and concretes are durable and leachates are low. Mineralogical analysis was not conducted on cured samples. Samples created showed significantly lower strengths than Portland cement samples.

**Rice Husk Ash**

Jaturapitakkul and Raosreung (2003) mixed Calcium Carbide Residue (CCR), a by-product of acetylene production (John 1993) and Rice Husk Ash (RHA; for every 1000kg of rice grains produced there is 200kg of rice husk waste, of which 85-90% is amorphous silica). CCR consists mainly of calcium hydroxide and is obtained in slurry form. RHA contains a high amount of $Si0_2$, most of which is in amorphous form (Gambhir, 1995; Mehta, 1986) which makes RHA a pozzolanic material in accordance with ASTM C 618 (1997d).

Kranmart et al (1996) used 30% of CCR and 70% of fly ash by weight as the cementitious material in mortar and obtained compressive strengths of 20.9 MPa at 90 days. The materials used were ground before being mixed into the samples. Mortar samples were made using varying quantities. A reference sample was made with Portland cement. 5 cm cubes were cast with the mixes and the compressive strengths of the mortars determined at the use of 1, 3, 7, 14, 28, 60, 90 and 180 days curing. Results showed that of the CCR and RHA mixes the 50:50 mixture gave the highest strength, which was 50% of the Portland cement reference samples at 28 and 180 days. The CCR-RHA samples also took longer to develop their

compressive strength. Juturapitakkul and Roonsreung (2003) concluded that CCR-RHA could potentially be used as a Portland cement replacement in low strength concretes, but stated that further development was required to achieve faster strength development and further testing of long term durability.

SEM analysis was conducted on initial CCR and RHA particles but unfortunately no analysis was conducted upon hydrated samples. However the materials used did not require any processing other than grinding and were used as a complete replacement for Portland cement.

Poon et al (2003) conducted a study comparing the hydration products and compressive strengths of mortars prepared with partial replacement of Portland cement with PFA and reject fly ash (rFA). Reject fly ash makes up a significant portion of Pulverised Fuel Ash (PFA) generated by coal fired power plants and is rejected due to its high carbon content and large particle size.

Poon et al (2003) found that compressive strengths were reduced in comparison to samples prepared with Portland cement. In addition to compressive strength testing. They conducted a very thorough programme of SEM analysis of hydrated mortars at 7, 28 and 90 days curing. This analysis showed that rFA particles only became significantly active after 28 days curing and that the addition of Portland cement increased the activity and formed hydrated cement products. They concluded that rFA was not as reactive as PFA or Portland cements and required longer curing periods to gain strength.

Dourdonounis et al (2004) investigated the effectiveness of high alumina cement production from FeNi-ERF slag limestone and diasporic bauxite ($2Na\ AlO_2$). Mortars produced samples that exceeded the compressive strength of Portland Cement mortars at 7 and 28 days. However, the FeNi-ERF samples were significantly weaker between 1 and 3 days. Dourdounis et al (2004) conducted mineralogical analysis on the FeNi-ERF slag cement and found many cement forming compounds to be present.

### 2.2.2 Use of Waste Aggregates

Obtaining sufficient aggregates for concretes is frequently a difficult and expensive problem, especially in areas such as south east England where topography and urbanisation make quarrying logistically difficult. Aggregates are traditionally won from alluvial deposits (natural aggregates) or from crushed rock. Whilst aggregates are generally low cost materials (transportation usually accounts for most of the cost) quarrying of both natural and crushed rock aggregates has significant negative environmental impacts. Because of this and the introduction of aggregate tax the construction industry has made efforts to recycle aggregates and to find sustainable sources of aggregates such as "wastes".

#### 2.2.2.1 Recycled Aggregates

Chen et al (2002) conducted research into the mechanical properties of concrete made with re-cycled rubble aggregate. They found that mortars and concretes made with re-cycled aggregate exhibited significantly lower compressive strengths and elastic moduli. However, washing the aggregate before use reduced this effect, and whilst the mortars and concretes would still have inferior mechanical properties, recycled rubble aggregates could still be used to make low strength mortars and concretes. Whilst their study of mechanical properties was thorough, Chen et al (2003) did not conduct any testing for the effects that the recycled aggregate had on the chemical and mineralogical properties of the cements and mortars.

Wirquin et al (2000) reported that a study of water absorption in recycled aggregate concretes showed that the processes of water absorption in re-cycled aggregate and in natural aggregate concretes are similar and obey the same laws. In addition, Metha and Monteiro (1994) reported that the water, as a primary agent, is able to create and degrade natural and artificial materials as concretes, implying that the use of re-cycled aggregates does not have detrimental effects on the initial stages of concrete mixing by sorbing the water needed for the hydration of the cement any more than natural aggregates do.

Levy and Helene (2004) analysed the durability of concretes made with 0, 20, 50, and 100% of aggregates from recycled sources. Durability was accessed by measuring water absorption, total pore volume and carbonation. Levy and Helene (2004) concluded that concrete made with partial or total replacement of aggregates with recycled aggregates can have the same fresh workability and can achieve the same compressive strength as concretes made with 100% natural aggregates. Of the parameters used to measure durability Levy and Helene (2004) found that samples made with recycled aggregate exhibited better behaviour than control samples made with natural aggregates. It should be noted that whilst Levy and Helene (2004) measured properties which are indicative of durability they did not conduct durability tests (e.g. ASTM D590 freeze thaw), which must be considered a more reliable method of measuring durability. Also, the mineralogical effects of using recycled aggregates were not assessed as part of this study.

#### 2.2.2.2 Aggregates from Industrial Sources

Glass in general is a highly transparent material formed by melting a mixture of materials such as silica, soda ash, and calcium carbonate ($CaCO_3$) at high temperatures followed by cooling during which solidification occurs without crystallisation.

Park, Lee and Kim (2004), investigated the potential of using crushed waste glass as a fine aggregate in concretes. They compared the chemistry and material properties of 3 different crushed waste glassess (amber, green and flint) as raw materials and their effects of the properties of fresh and cured concretes. Once crushed, the waste glass had a grain size of between 6-20mm and angular grain shape. Waste glass was used to replace natural aggregates in 30%, 50% and 70% amounts. They found that slump was reduced with increasing glass content. This they ascribed to the angular nature of the glass with cement paste attaching to the glass thus reducing the amount of water available for fluidity. Compressive strengths were reduced in cured samples with increasing waste glass content. However, with admixtures compressive strengths still exceeded 25 $N/mm^2$. Park et al (2004) concluded that, provided glass content was kept below 30% and a suitable admixture was used to

secure fluidity when mixing, glass could be used as an effective aggregate. Park et al (2004) did not conduct any mineralogical analysis of the cured concrete.

Lightweight aggregate is an important material in reducing the unit weight of concrete used for special concrete structures of large high rise buildings. Generally, lightweight aggregate is made from ground granulated blast furnace slag (GBFS), fly ash, and volcanic ash (ACI, 1994; Neville, 1996). However, the production and use of lightweight aggregate faces some problems.

- The high cost of aggregate due to high incineration temperature.
- The shrinkage and the resistance to freezing and thawing because of high absorption water. (Kohao et al, 1999 and Hon et al, 1998).

Waste polyethylene terephthalate (PET) bottles are produced in great number around the world. PET bottles were initially reworked to produce more drinking bottles by melting fusion, but this proved to be too expensive. Processed PET bottles were then trialled as lightweight aggregates, but initial results were unsatisfactory. However Choi et al (2004) found by adding Ground Granulated Blastfurnace Slag (GGBS) to shredded PET bottles, whilst being mixed and melted to form lightweight aggregate pellets, the GGBS formed a skin around the PET aggregate which bonded cement pastes to the untreated PET aggregate. Despite finding that replacing conventional aggregate with the PET aggregate reduced the strength and workability of the concrete, it was still possible to make a lightweight concrete with adequate properties. Choi et al (2004) conducted SEM analysis on cured concrete samples and verified the integrity of the interface between the PET aggregate and the cement paste. Choi et al concluded that concrete with 70% replacement PET aggregate was only 21% less structurally efficient than other lightweight aggregate, and with the right treatment would make an acceptable lightweight aggregate.

Pera et al (2004) investigated the use of valorised automotive shredder residue in building materials. It has been identified that, within the EU nations, 12 million cars are disposed of per annum. 75% of the material generated is metal that can be

recycled (70% iron and steel, 5% non ferrous metals). The remaining material, called automotive shredder residue (ASR), accounts for about 3 million tonnes of material being landfilled every year in Europe. Some of the ASR has been utilised (after processing) into an alternative fuel (Ref 1-3), although a large proportion, called incombustible shredder residue (ISR) has yet to be proven to have an alternate use.

Pera et al (2003) conducted experiments in which ISR was calcined then immersed in sodium hydroxide solution or sulphuric acid, followed by casting in Portland Cement mortar, and then directly mixed with calcium sulfoaluminate cement without pre-treatment. Pera et al (2003) found that similar compressive strengths to a control mix could be achieved with both techniques, but that setting times were retarded significantly.

Eldin and Senouci (1993) investigated the strength and toughness of concrete with a portion of aggregates replaced by waste tyre chips. They observed that the compressive strength and split tensile strength were reduced, while its toughness and ability to absorb fracture energy were enhanced significantly. Topcu (1995) investigated the effects of particle size and content of tyre rubber on the mechanical properties of concrete. He found that, although the strength was reduced, the plastic capacity was enhanced significantly.

Li et al (2004) investigated the use of tyre chips and fibres in concretes. Compressive strength, compressive modulus of elasticity, Poisson's ratio and split tensile strength tests were conducted on prepared samples to ASTM standards. They concluded that tyre fibres perform better than tyre chips and that fibre length should be further investigated. Steel wires contained within the fibres and chips had a positive effect on strength and stiffness properties. Truck tyres performed better than car tyres. The overall strength of concretes was reduced when chips and fibres were added, but however, the brittleness of the concrete was reduced. The use of tyres in concrete can be an effective method of disposal as concrete of an acceptable standard can be produced in this way.

### 2.2.3 Wastes in Ground Improvement

The use of waste materials in ground improvement (treatment/stabilisation of contaminants, strength improvement etc.) technologies is less common than in cements and concretes although some research has been conducted and applications devised.

Certain materials previously considered to be "waste" products have been investigated as chemical binders because of their capacity to sorb various contaminants and their low costs.

Examples of such materials which have been tested include granulated tyre, wood shavings, straw, used peat, dried sewage sludge, leaf tea and tree leaves. These materials sorb different contaminants. For example tyre is effective in sorbing hydrocarbons and has been shown to sorb up to its own volume (Al-Tabbaa, 2000). Wood shavings, straw and peat are effective in sorbing copper with up to 80% sorption being observed (Al-Tabbaa et al, 2001 b). Preliminary investigations in which these "waste" materials have been used as part of a cementitious binder system have shown that their sorption capacity is not affected by the presence of a cementitious matrix.

#### 2.2.3.1 Waste Tyres

Yoon et al (2004) conducted research in which they made mats from tyre treads and sidewalls, then placed them in layers in loose sand. They then conducted a series of plate loading tests. Sand reinforced by waste tyres had more than twice the bearing capacity of untreated loose sand, although this decreased with increased sand density. Settlement reduction due to tyre reinforcement with a combination of treads and sidewalls was as much as 70% for loose sand and 34% for dense sand. Whilst this use could prove a potential alternative to disposal in landfill, processing of the waste tyres for use in this form of ground treatment would be labour intensive and would use relatively few tyres.

### 2.2.3.2 Waste Gypsum

Many researchers have observed that when waste gypsum is added to lime ($Ca(OH)_2$) a new binding agent called gypsumlime is formed, which improves the strength development of clays in deep stabilisation, Kujala (1982), Neiminen (1978).

Kujala and Niemen (1983) conducted tests using waste gypsum formed as a bi-product of the production of concentrated fertilizers when phosphoric acid is made from apatite and sulphuric acid. The waste gypsum is calcium sulphate containing water of crystalisation ($CaSO_4.2H_2O$). Kujala and Niemen (1983) mixed the waste gypsum in a 1:1 ratio with unslaked lime (calcium oxide, CaO). They found that the composition of the gypsumlime varied considerably when mixed in different conditions (large or small batches etc.) and that the performance of the gypsumlime as a soil binder was affected by these differences. The gypsumlime binder was mixed with a number of clay soils and the modified soils were studied by mineralogical structural analysis. Results showed the formation of ettringite and calciumsulphoaluminate within the soils. Analysis of both field and laboratory samples showed that greater strengths could be obtained with gypsumlime than with unslaked lime alone. Kujala and Nieminen concluded that the reaction between the binding agent (gypsumlime) and basic constituents of the soil is always chemical in nature and thus the final result depends essentially on the composition of the initial materials. Unfortunately Kujala and Nieminen did not present the results of the field and laboratory investigations of soil strength and did not reference any other documents that may contain this information so the strengths obtained are not known. Studies of gypsumlime were also conducted by Kujala (1983), who mixed waste gypsum with unslaked lime and added to a number of clay soils. Reference samples were made mixing the same soils with unslaked lime. A series of laboratory and field tests were conducted on the stabilised soil. The results of soils laboratory triaxial tests showed that gypsumlime was more effective than lime on its own and developed strength faster. For the in-situ tests an embankment was constructed over a foundation one half lime/one half gypsumlime columns. Settlement was found to be the same on either side of the embankment after two loading stages. Kujala (1983) attributes the reason for the difference between

laboratory and field results as being caused by less efficient mixing. He concluded that this had reduced the quantity of etteringite formed and expected in the longer term, after 3 loading stages, that the gypsumlime section of the embankment would exhibit less settlement than the lime section. However, this was not demonstrated by any results.

Tonks et al (2000) identified the potential of using waste anhydrite as a temporary site road surface. When dry anhydrite powder was soaked and lightly compacted, a hard surface could be made achieving the strength of low grade concrete after 28 days and a permeability of 6-9 $x10^{-9}$ m/s. Mineralogical analysis showed that around 50% of the anhydrite converted to gypsum during a period of 28 days. The gypsum crystals partially filled the pore spaces within the samples thus increasing strength and reducing permeability.

Holm et al (1983) used gypsum in combination with lime in 50:50 and 75:25 mixes although it is not made clear in their paper whether the gypsum is a waste product. It could have been sourced as an industrial by-product. Holm et al (1983) conducted a trial in silts and clays with undrained shear strength of between 10 and 20 kPa from Hoddige, Sweden. Laboratory and in-situ tests were carried out on a range of silts and clays from the trial area. In both field and laboratory tests Holm et al (1983) found that a mixture of gypsum and lime (slaked, calcium hydroxide $Ca(OH)_2$ was considerably more effective as a binding agent than lime alone, with the same shear strength being reached in half the time. Although it is interesting to note that shear strengths from the laboratory samples tested at curing periods over 100 days dipped slightly before increasing again when tested at 1000 days. Holm et al (1983) concluded that when high strengths are required over a period of several years a mix ratio of 75:25 lime: gypsum is best as this generates higher strengths in the longer term and are believed to be more durable when long term pH is taken into account. Holm et al suggested that ettringite formed in the gypsum-lime mixes would only be stable so long as pH levels remained high. Therefore the quantity of lime within the binder should be optimised.

#### 2.2.3.3 Ashes

The use of coal ash as a soil binder for use in road construction has been investigated by Tsonis et al (1983) when mixed with lime and cement. The ash tested was taken from a coal burning power station in Magalopolis, Greece. Processing of the ash was required before use in that it had to be pulverised in order to crush larger lumps which form during stockpiling.

Ash was added to two soils, silty clay and sand, at 35% by dry weight, although no details of the index properties of these soils are presented. Lime or cement was then added to the samples in 1% increments. Samples were prepared at optimum densities as calculated from compaction tests, and tested for compressive strength. strengths of up to $2MN/m^2$ were achieved in some mixes, although poor presentation of results does not clearly show whether strength benefits are derived from the addition of lime, cement or the ash used. Tsonis et al (1983) concluded that lignite ash from Magalopis power station could be utilised as a soil mixing agent for preparation of improved sub-grade or sub-bases.

The Tsonis et al (1983) study tested an interesting concept, but is badly let down by poor reporting of soils tested, poorly explained and confused testing philosophy, and confused presentation of results. This is very unfortunate as the study had the potential to prove the viability of "waste" material as a low cost soil binding agent.

### 2.2.4 Wastes in Ground Remediation

#### 2.2.4.1 Contaminated Land

PFA-lime products containing waste possess favourable leaching characteristics, especially for wastes containing heavy metals, where the metals ions may be chemically bound to the hydrate complexes (Al-Tabbaa and Perera, 2001). It is thought that the unburned carbon content in PFA generally acts as a sorbent for certain wastes including organics (Barth et al, 1990; La Grega et al, 1994). However, PFA-lime solidified waste products are less durable and have higher

leaching rates than those containing cement (Harris et al 1995a). PFA can be used together with cement, with the amount of cement replacement dependent upon the requirement of the end product.

PFA may be suitable for the stabilisation of both inorganics and organics. Contaminants such as hydrocarbons from oil exploration can affect the environment and the safety of civil engineering structures (Preslo et al, 1989; Nicholson and Tsugawa, 1996; Shroff et al, 1989). Cleaning contaminants from soil can be a complicated task by virtue of high cost; disposal of contaminated soils is also expensive.

Shah et al (2002) conducted a trial to stabilise soil contaminated by petrochemicals at Undhera Village, Vadodara District, India. They mixed a cementitious binder composed of lime, fly ash and cement mixed in various proportions at three different weight percentage additions (5, 10 and 20%). The contaminated soil was a loamy silt with percentage by weight of oil between 7-10%. Index properties of contaminated and uncontaminated soil were tested and it was found that maximum dry density, optimum moisture content, unconfined compressive strength (UCS), cohesion and angle of internal friction were all reduced in the contaminated soil. Once the contaminated soil had been mixed with the binder, UCS tests, permeability, leachate, X-ray diffraction, SEM analysis and Plasticity Index (PI) tests were conducted on the mixed samples. The addition of the binder improved the shear strength of the contaminated soil from a shear strength of 0.38 kPa to strengths up to 138 kPa. Although curing periods are not mentioned in the paper increases in ǿ (effective angle of internal friction) and c' (effective cohesion) were also noted, whereas permeability and leachability of the soil were decreased.

X-ray diffractometry of treated and untreated soil (10% lime, 5% fly ash and 5% cement) was carried out to evaluate the formation of mineral/chemical compounds due to the action of stabilisation agents. Analysis indicated the possible presence of Calcium Silicate Hydrate, Calcium Alluminate Hydrate and Calcium Alluminate Silicate Hydrate. SEM analysis indicated that the addition of the stabilisation admixture (10% lime, 5% fly ash and 5% cement) resulted in the formation of non-crystalline chemical compounds.

The study shows that stabilisation of hydrocarbon contaminated clay soils is possible using a cementitious binder with a waste (fly ash) element. The XRD and SEM investigation is a good attempt to assess the chemical/mineral compounds formed by the addition of the binder but it should be noted that concluding that CSH, CAH and CASH have formed on the basis of XRD results may not be possible as these compounds are X-ray amorphous as they do not form crystals. This is confirmed by the fact that Shah et al (2002) state that SEM analysis indicates the presence of "non crystalline chemical compounds". It is also unfortunate that curing details are not given in the paper.

#### 2.2.4.2 Abandoned Mine Workings

Abandoned mine workings pose a significant risk where structures are built over them because of possible subsidence caused by the collapse of workings. The use of PFA cement and water mixtures is an established technique of filling the old workings voids with low viscosity fast strengthening grouts to stabilise the mine workings and preventing collapse.

However the treatment of mines with large cavities makes grouting excessively expensive due to material costs of gravel and cement. Using a greater proportion of PFA in grout usually reduces the cost as stockpiled fly ash can usually be obtained for only a little over the cost of transportation.

Jarvis and Brooks (1996) conducted a project investigating the use of higher proportions of PFA in the grouts. They tested a number of PFA pastes with cement controls of between 1 and 5%. PFA was sourced from a number of different stockpiles. 100mm cubes were prepared from the mixes and tested at 3, 7, 28 and 90 days. Grouts were also flow tested to ensure they could be easily pumped. Cube strengths obtained were around 1000 kPa at 7 days. Higher cement contents yielded higher strengths, which continued to increase up to 90 days. As expected higher water contents produced higher flow rates. However, cubes prepared with low cement content (<1%) displayed considerable variability in strengths.

From results obtained from UCS cube testing, pastes were designed for the stabilisation of two mines, (Castlefields and Cow Pasture), both located in the west midlands of England. At the Castlefields Mine a grout with 2% cement content was used with a 28 day cube strength requirement of 200kPa. At Cow Pasture mine a grout with 4% cement and a 28 day cube strength requirement of 1500 kPa was used. Satisfactory flow was achieved when placing the grouts. In-situ testing indicated that strength requirements were achieved. Jarvis and Brooks concluded that provided pastes are properly designed and quality control methods maintained, then low cement PFA pastes are suitable to be used in the stabilisation of mine workings. However, mixes should not be made with less than 2% cement as variable strengths are produced. The study proves that economical pastes can be produced from PFA. It would have been useful if a more thorough mineralogical investigation had been conducted as part of the investigation.

### 2.2.5 Case Studies

Soil stabilisation of soft soils through soil mixing techniques is well documented, e.g. Lin and Wong (1999), Bergado et al (1999); however, the successful treatment of organic soils has been less well documented. Most of the data available on soil stabilisation projects relate to the stabilisation of soft clays containing small amounts of organic matter. Kuno et al (1989) showed from results of tests conducted on very soft soils from Japan that the humic acid content greatly influenced the soil improvement effect generated by the binder mixed with the soil.

Hebib and Farrell (2003) conducted a study to assess the effectiveness of using combinations of cement, PFA, lime, blast furnace slag, blast furnace slag and cement, and blast furnace slag and gypsum to stabilise peat soils with very low shear strengths. Binders were added at 150, 200, and 250 kg/m$^3$ of soil. Pure cement was used as a control.

Samples were prepared by mixing in a laboratory dough mixer in a dry state. These samples were then immersed in water in plastic sample tubes under a confining pressure of 18kPa, representing the 1m of fill normally laid out on top stabilised columns in the field.

Unconfined compression strength tests were carried out after, 7 and 28, 90 and 365 days curing time. Triaxial compression tests were carried out at 90 days. Tests were also conducted in a specially made large testing chamber (1.68 diameter), and $1m^3$ peat block samples were taken for testing in this large chamber. A stabilised column (600mm in diameter) was created inside the chamber to simulate in-situ conditions. Hebib and Farrell (2003) concluded that the strength of the material stabilised with cement was significantly higher than untreated peat. When PFA and lime were used as the stabilisation binder the increase in strength was low, but when gypsum was added to the binder the increase in strength was made much greater for one of the two peats tested. The same results were obtained when blast furnace slag was used as the binder. Inspection of the cement peat mixtures under the electron microscope indicated the cement particles filled the void spaces in the peat, but no interaction between the hydrated cement products and the peat was observed (although no mention is made of electron microscope work elsewhere in the paper). It is also unfortunate that no mention is made of microscope work done on samples of peat mixed with other binders. Permeability of the treated samples was found to be the same or lower than untreated samples and dependant upon preloading. Samples that had been pre-loaded yielded lower permeabilities and higher strengths.

Al-Tabbaa and Evans, (1998), Al-Tabbaa et al (1998), Al-Tabbaa and Evans (2000) and Al-Tabbaa and Boes, (2002) conducted a study to develop soil-grout mixes and equipment suitable for solidification / stabilisation of soils at a site in West Drayton UK. They presented their research findings in 4 papers published between January 1998 and July 2002. These publications concentrated on

a) laboratory study to access potential grout mixes
b) the development of a prototype mixing auger
c) site trial
d) long term performance.

In part 1 of the laboratory study, various combinations of cement, PFA and lime in various ratios were tested as grouts. The soil to be stabilised was made ground composed of sands and sandy clays plus natural sands and gravels. The soil was from the site of a former chemical works, and as such contained a wide range of contaminants including flammable materials, solvents, concentrated sulphuric acids, oils, petrol, radioactive materials, and other unidentified materials. Al-Tabbaa and Evans (1998) note that "Representative samples from each of the soil types were classified by sieving the granular fraction of the soils only". In the case of the made ground 40% of the soil mass fell into the silt clay fraction, but it is unfortunate that the ratio of silt to clay has not been investigated as the clay content of the soils would be expected to affect the effectivness of the solidification of the soil.

A range of tests were conducted upon the stabilised samples. Wet-dry and freeze-thaw (ASTM D559 and D560) were conducted on samples cured for 28 days samples. All mixes survived all 12 cycles with a maximum percentage dry mass of less than 4%. All the mixes failed the freeze-thaw test, the general mode of failure in all cases being the development of vertical cracks along the full length of the samples. As would be anticipated the mixes which contained more cement survived longer. Al-Tabbaa and Evans (1998) pointed out that the freeze-thaw test is not representative of the conditions in the UK and that less stringent conditions would be more appropriate.

UCS tests were conducted on the samples originally wet-dry tested. Results showed that the samples had not lost any strength.
Permeability testing showed that samples had permeability in the region of $x^{10-8}$ – $x^{10-9}$ m/s.

The study concluded that 4 soil grout mixes should be selected for further study, containing 4% cement and 17% grout. The mixes produced 28 days UCS values of between 350 and 1100 kPa, densities between 1500 and 1850 kg/m3, leachate pH values between 6.5 and 10.5, plus satisfactory wet-dry and poor freeze thaw durability performances. An additional 3 mixes were devised for further study (see Table 2.1 for grout details of all 7 mixes)

Table 2.1, Mixes (after Al-Tabbaa and Evans, 1998)

| Mix | Cement:pfa:lime:bentonite | Water:dry grout | Soil:grout | Soil:dry grout |
|---|---|---|---|---|
| A | 2 : 8 : 0 : 0 | 0.42 : 1 | 5 : 1 | 7 : 1 |
| B | 3 : 8 : 0 : 0 | 0.42 : 1 | 5 : 1 | 7 : 1 |
| C | 2.5 : 8 : 0.4 : 0 | 0.42 : 1 | 5 : 1 | 7 : 1 |
| D | 3 : 8 : 0.1 : 0 | 0.42 : 1 | 5 : 1 | 7 : 1 |
| E | 2.5 : 8 : 0.4 : 0 | 0.42 : 1 | 3.5 : 1 | 5 : 1 |
| F | 2.5 : 8 : 0.4 : 0 | 0.30 : 1 | 3.9 : 1 | 5 : 1 |
| G | 8 : 0 : 0 : 0.8 | 1.6 : 1 | 2.8 : 1 | 7.3 : 1 |

In the second part of the study the development of the auger used is detailed along with results of the site trial.

The seven grout mixes from the previous study (see Table 2.1) were mixed into overlapping columns using the prototype auger. Al-Tabbaa and Evans reported that, aside from minor difficulties associated with ground conditions, the grout columns were installed successfully. Following installation the columns were left to cure *in-situ* and cores were then taken from the columns after 28 and 45 days. The treated ground was then excavated for visual inspection. The columns were easily distinguishable from untreated ground and there were no obvious planes of weakness.

Unfortunately the recovered samples did not have uniform diameters. However, Al-Tabbaa et al (1998) took this into account when calculating test results but they note that samples tested for unconfined compression strength had diameter : length ratios between 1:2 and 1:1, (a consistent 1:2 ratio would have been more desirable). In addition to UCS tests, Al-Tabbaa et al (1998) conducted durability and permeability tests on recovered samples.

Al-Tabbaa et al (1998) concluded that the prototype auger was successful in installing soil-grout columns with the selected mixes. 60-70 day made ground cores produced UCS values between 950 and 1500 kPa, leachate pH values of between

9.5 and 11, satisfactory wet-dry and modified freeze thaw durability, permeability between 0.5 and $3x10^{-9}$ m/s, and acceptably low compressibility. These properties were generally better than those achieved in the earlier treatability study. Al-Tabbaa et al (1998) ascribed this improvement to better compaction being achieved in-situ although normally strengths achieved in-situ are lower than those seen in the lab.

Al-Tabbaa et al (1998) do not mention in-situ testing of the columns in their paper. This would have been a useful addition to the study as it might have given more insight into variable strengths achieved within the columns. It is acknowledged that in-situ testing can cause slight damage to stabilised columns and this may have been undesirable in the long term.

Also mineralogical testing was not included in the programme. It would have been interesting to run a comparison of samples cured in-situ and in laboratory conditions to see if there were mineralogical reasons for the differences in the strength.

In Part 3 of the study Al-Tabbaa and Evans (2000) tested cores at 14 and 28 months, these samples having been initially cured in-situ for 45 days, the remaining curing took place in the laboratory. Samples were subjected to UCS, durability (wet-dry and freeze thaw), permeability, compressibility and leachate pH tests. R-ray diffraction and scanning electron microscope tests were also conducted on the 14 month old samples.

From this stage of results Al-Tabbaa and Evans (2000) concluded that samples at 2 months typically had unconfined compressive strengths of 1000-1500 kPa, and this increased to 3200 kPa at 28 months. Negligible deterioration was noted in wet-dry durability tests. Permeability of $0.6 \times 10^{-9}$ to $3 \times 10^{-9}$ m/s reduced to $0.1 \times 10^{-9}$ m/s to $0.8 \times 10^{-9}$ m/s at 14 months, then increased to $0.2 \times 10^{-9}$ to $1.3 \times 10^{-9}$ at 28 months, and similar results were recorded for volume compressibility. Leachate pH continued to decrease from 9.5 to 10.9 at two months, to 6.7 to 8.5 at 28 months. The X-ray diffraction analysis at 14 months identified limited crystalline phases normally associated with the hydration of cementitious materials. However, scanning electron microscope analysis indicated the presence of many of the

characteristic phases (C-S-H, C-A-H, CH and ettringite). This is understandable as many of the mineral phases produced in cement hydration reaction are only poorly crystalline, or X-ray amorphous, and would not show up in the analysis.

In Part 3 of the study cores drilled at 45 days were tested after being stored in the laboratory. In this part of the study the soil grout columns were re-cored after 5 years of in-situ curing.

In Part 4 of the study (Al-Tabbaa and Boes, 2002) the performance of the soil grout mixes was evaluated at 5 years. Recovered cores were again subjected to a suite of tests including UCS, permeability, wet-dry and freeze thaw durability, leachability, plus scanning electron microscopy and X-ray diffraction analysis. Across the range of tests there were strong indications that hydration of the columns was still taking place.

The original design criteria were being met and overall the study proved that cement : PFA : lime : bentonite grout mixes were effective in solidifying / stabilising contaminated ground. The authors intention is to conduct further tests at 10 years curing. Overall the study has yielded high quality data on a technique only fairly recently introduced to the UK. The quality and quantity of mineralogical work is particularly impressive.

## 2.3 Potential for Use of Wastes and Co-products in Construction

### 2.3.1 Identification of Potential Uses

The manner in which wastes and co-products can be used within the construction industry largely depends upon the nature of the waste in question. A large number of ashes have been shown to be suitable as cement replacements or as aggregates. Larger inert wastes (waste tyres, plastics, glass) have also been proven to have potential as aggregates. This thesis considers potential uses of co-product Red Gypsum and as such the properties of the Red Gypsum are critical in finding potential engineering applications.

Grant (2000), suggested various engineering/disposal options for Red Gypsum. However, the options listed amount to disposal in landfill and all would incur disposal costs.

Red Gypsum has previously been trialled as a fill material (Grant, 2000). Such an application is only cost effective when the material is used close to the site of production due to the cost of transportation. It would therefore be more desirable to make use of other properties of the material (i.e cementitious etc.).

Red Gypsum is already used as an agricultural soil conditioner because of its pH properties. This application could be developed further. Material could potentially be mixed with contaminated land, spoil mounds and tailings lagoons to facilitate remediation and re-vegetation.

It has been observed (Anon, 2001) that Red Gypsum increases the Plasticity Index of clay soils. This property has the potential to be exploited in two ways. Firstly, by mixing Red Gypsum with soil in temporary haul roads where the resultant increase in Plasticity Index makes the soil less sensitive to water logging and rutting damage from heavy vehicle traffic. Plasticity Index also affects the susceptibility to shrink and swell due to seasonal water content variation (Craig, 1992). Red Gypsum could potentially be added to such soils to modify the Plasticity Index and hence reduce the sensitivity to shrink and swell.

Studies conducted at Newcastle University (Anon, 2001) indicated that when mixed with other materials Red Gypsum developed high strengths and stiffness, blast furnace slag proving particularly effective. Within the field of ground improvement there are many potential uses for cementitious materials as has been shown in previous sections. Drying and grinding Red Gypsum filter cake and mixing it with other materials could produce a soil mixing binder suitable to be used as a surface or deep dry soil mixing binder. Unprocessed filter cake could be mixed with materials like blastfurnace slag and used as a shallow soil binding agent. Cementitious grouts could also be produced from Red Gypsum based binders.

### 2.3.2 Sources of Material

As covered in the previous sections, there are many potential sources of waste materials that could potentially be more widely utilised in the construction industry. Some of these have already been trialled in some applications. Pulverised Fuel Ash and Ground Granulated Blast Furnace Slag are waste derived materials, and as previously explained in this Chapter, have already been used in civil engineering applications. However, there is scope for further exploitation of these materials. Whilst it is acknowledged that there are many other materials that have the potential to be utilised in construction, this study is focussed on the use of waste industrial gypsum. Other waste derived materials used in the study are described in Section 2.4.

Beretka et al (1996) identified the fact that industrial gypsums are particularly under utilised. The world wide production of phosphogypsum generated per anum is about 150 million tonnes, but on average its utilisation rate does not exceed 15% of output, (Carmichael, 1986). The largest source of waste gypsum derives from the manufacture of Phosphoric acid, by reaction of Phosphate rock with sulphuric acid (UN, 1988).

Other sources of gypsum include gypsum produced during the manufacture of Titanium Dioxide and desulphogypsum, produced during the process of desulphurisation within coal fired power stations (Boari et al, 1992).
The production of phosphate fertilisers generates waste gypsum when phosphoric acid is made from apatite and sulphuric acid is used in the process. The waste gypsum is calcium sulphate.

## 2.4 Certification/Legislation of New Construction Products

### 2.4.1 Introduction

Certification of new building materials in the UK is carried out by BRE Certification, a sister company to the Building Research Establishment (BRE) which carries out research into the safety, regulation and performance of buildings

on behalf of the UK Government. BRE Certification is a wholly owned subsidiary of the Foundation for the Built Environment which is a non profit distributing organisation. The Loss Prevention Certification Board joined BRE Certification on 31st March 2000, and WIMLAS joined BRE Certification in June 2000. WIMLAS (formerly part of Wimpey Laboratories) has carried out assessment of construction products for the last 50 years.

### 2.4.2 Approval

Approval (certification) is third party confirmation that products, services, systems and personnel meet, and continue to meet, certain standards and specifications. Approval may be required for many reasons including:

- Legislation
- Politics
- Differentiation
- Life safety
- Property protection
- Commission directives
- Risk management

BRE Certification offers approval schemes for products, processes, personnel, quality systems, environmental management systems and safety management systems. They are also a Technical Approvals issuing body offering approvals of products under the Building Regulations, Construction Products Directive and other European Legislation. Approval or certification schemes are designed to meet specification needs and may involve the preparation of specific standards. It is also possible to approve individual products based on a product specific risk assessment and performance specification.

### 2.4.3 Technical Approval Certificates

Where published standards do not exist, BRE Certification can issue Technical Approvals which meet the requirements of relevant legislation such as Building Regulations or directives and insurance requirements.

### 2.4.4 Accreditation

BRE Certification are accredited by the United Kingdom Accreditation Services (UKAS) against the following standards.

EN 45011 – product certification
EN 45012 – management systems certification
EN 45013 – personnel certification

### 2.4.5 Construction Products Directive

From the 1st of April 2001 it has been possible for the construction products to be placed on the UK and European Economic Area (EEA) market with CE marking based on a harmonised European Standard (Construction Products Directives 89/106/EEC). This directive provides for common methods of performance evaluation of the product across the EEA and these methods are described in the relevant EN standards that are being progressively published over the next 4 years. Products which are covered by the Construction Products Directives (CPD) are those which are "produced for incorporation in a permanent manner in works". In this case "works" include buildings, roads, bridges and other civil engineering and building works.

The Directive also contains essential requirements for the performance of works. These are:

- Mechanical resistance and stability
- Safety in the case of fire
- Hygiene, health and the environmental
- Safety in use
- Protection against noise
- Energy economy and heat retention

## 2.5 Identification of Materials Investigated in This Project

### 2.5.1 Blast furnace Slag

Blast furnace slags are obtained from the manufacture of pig iron and contain silica, alumina and lime (Neville and Brookes, 1993). They are different from pozzolans in that the nature of the reactions and the reaction products are different (Harris et al, 1995a). Ground granulated blast furnace slag (GGBS), which is the type most available in the UK, is classed as a latent hydraulic cement with compositions broadly intermediate between pozzolanic material and Portland cements (Taylor, 1990).

The hydration of slag is initiated when lime provides the correct alkalinity, but subsequent hydration does not rely on lime. Reactivity depends on factors such as bulk composition, glass content and the fineness of the grinding, and the relationship between composition and glass content is quite complex (Taylor, 1990).

GGBS is available as a separated ingredient to be added to treatment systems at the point of mixing, either alone or with other binders, and as blends in various proportions with Portland cement. These can be used as partial replacement for cement and bring about cost savings.

### 2.5.2 Pulverised Fuel Ash

Pulverised Fuel Ash (PFA) is a synthetic pozzolana created by the combustion of coal. There are two main types of PFA in existence, namely low lime PFA and high-lime. UK ashes are generally classified as low lime PFA. The material consists mostly of glassy, hollow, spherical particles called cenospheres. PFA can be described as a siliceous and aluminous material which on its own possesses very little cementitious properties, but if finely divided and mixed with water will react chemically with lime to form compounds possessing cementitious properties (Neville and Brookes, 1993). However, not all types of PFA exhibit good pozzolanic properties (Harris et al 1995a). The reactivity appears to depend upon

the nature and proportion of the glass phase present (Dhir, 1986). The stabilisation effect of PFA relies on the formation of calcium silicate gels which harden over a long period to form a stable material (Harris et al 1995a). Despite being slow to harden the hydration products may be similar to those of OPC (Taylor, 1990).

### 2.5.3 Synthetic Red Gypsum

As previously stated (Chapter 1) Synthetic Red Gypsum is a by-product of the production of titanium dioxide ($TiO_2$) pigment. Titanium dioxide can be produced by two different processes, the sulphuric acid (or sulphate) process or the chloride process. In current practice several manufacturers (including Huntsman Group) operate both processes; however, only the sulphate process generates the co-product gypsum. In order to make 1 tonne of titanium dioxide pigment the sulphate process can produce several tonnes of co-product gypsum.

The production of titanium dioxide ($TiO_2$) pigment by the sulphate process uses two raw materials:

- Ilmenite
- Titanium Slag

Ilmenite is a natural ore, occurring in the form of a black sand or rock with the basic chemical formula $FeTiO_3$. Normally the iron is partly oxidised to the trivalent state and there are also significant siliceous impurities, Grant (1997). The $TiO_2$ content of ilmenite varies from 43 to 65%, Harden and Bates (1990). Titanium slag is also important; it is an enriched ore residue from the extraction of iron from ilmenite or mixed ilmenite-hematite/magnetite deposits. Harden and Bates (1990) list the $TiO_2$ content of titanium slag as between 70 to 74%, and occasionally as much as 85%.

The sulphate production process essentially comprises a series of simple chemical reactions.

$$Fe\ TiO_3 + 2H_2SO_4 \rightarrow TiOSO_4 + FeSO_4 + 2H_2O$$

$$TiO\ SO_4 \rightarrow TiO_2\ n\ H_2O + H_2SO_4$$

$$TiO_2\ n\ H_2O \rightarrow TiO_2 + n\ H_2O$$

The ilmenite and titanium slag are dried and ground then digested with sulphuric acid. This mixture is then agitated by compressed air and superheated steam. When a temperature of 100°C is reached an exothermic reaction starts and the mixture is converted to a porous cake containing ferrous, ferric and titanium sulphates. The cake is converted to water soluble sulphates by dissolving in water or dilute acid to form a black liquid, and the $TiO_2$ is recovered by hydrolysis of high $TiO_2$ solutions. Harden and Bates (1990) estimated the tonnes of raw material required to produce one tonne of $TiO_2$ as follows, illmenite or titanium slag 1.5-2.8 tonnes, sulphuric acid 3.0-4.0 tonnes, iron scrap 0.1-0.2 tonnes. Although older, the sulphate process remains popular due to the lower cost of raw materials but leads to a greater quantity of waste being produced.

The sulphate method of $TiO_2$ production generates an acidic effluent in the form of spent sulphuric acid. This waste liquid can either be concentrated and reintroduced to the pigment production process, or it can be neutralised. The high energy costs involved in concentrating the acid waste means that it is usually neutralised.

Neutralisation of the acid effluent is achieved in two phases which generate co-product gypsum. In the first phase the effluent is neutralised by the addition of calcium carbonate (limestone) which precipitates synthetic white gypsum slurry.

$$H_2SO_4 + CaCO_3 + H_2O \rightarrow CaSO_4 . 2H_2O + CO_2$$

The slurry is thickened by hydrocycloning then filtered and dried. Synthetic White Gypsum in the second phase overflow from the hydrocycloning is neutralised further to precipitate synthetic Red Gypsum, using either calcium carbonate or calcium hydroxide. The addition of calcium carbonate followed by aeration raises the pH from 6.5 to 7, resulting in a fully oxidised slurry with the following reaction chemistry.

$H_2SO_4 + Ca\ CO_3 + H_2O$ -> $CaSO_4 . 2H_2O + CO_2$

$4FeSO_4 + 10H_2O + O_2$ -> $2FeO_3 . 3H_2O + 4H_2SO_4$

$4FeSO_4 + 4CaCO_3 + 14H_2O + O_2$ -> $4CaSO_4\ 2H_2O + 2FeO_3 . 3H_2O + 4CO_2$

The resultant gypsum contains hydrous hematite ($2Fe_2\ O3 . 3H2O$). In contrast the addition of calcium hydroxide (slaked lime) raises the pH to 9.0 resulting in an unoxidised slurry with a different reaction chemistry.

$H_2SO_4 + Ca\ (OH)_2$ -> $CaSO_4 . 2H_2O$

$2H_2O + FeSO_4 + Ca(OH)_2$ -> $Fe(OH)_2 + CaSO_4 . 2H_2O$

In this case the gypsum contains iron as ferrous hydroxide, $Fe\ (OH)_2$ or iron hydroxide gels. In either case the resultant slurry is filtered and passed into solid filter cake composed of calcium sulphate and iron with traces of other metal hydroxides (see Table 2.2).

Table 2.2, Composition of red gypsum (after Peacock and Rimmer, 2000)

| Component | Content (by dry weight) |
|---|---|
| | % |
| $CaSO_4.2H_2O$ | 58.5-59.3 |
| $Fe_2O_3.H_2O$ | 32.9-36.6 |
| Ti | 1.0-1.3 |
| Al | 0.1-0.8 |
| Mg | 0.5-0.6 |
| Mn | 0.2-0.5 |
| Si | 0.-0.5 |
| Cl | 0.002-0.2 |
| | mg $kg^{-1}$ |
| Cr | 500-800 |
| Zn | 200-400 |
| Sr | 100-300 |
| Ni | 50-60 |
| Co | 20-30 |
| Ba | 1-3 |
| Pb | 1-2 |

### 2.5.4 Steel Slag

Steel slag is a by-product of the steel production process. Steelworks slag can be divided into two main types in accordance with their method of production, i.e. Basic Oxygen Steelmaking Slag (BOS); and Electric Arc Furnace slag (EAF). Typical chemical compositions of steel slags are shown in Table 2.3. The main mineral phases of BOS and EAF slags are dicalciumsilicate, dicalciumferrite and Wustite (Geisler et al 1992). The steel slag used in this study was ground weathered steel slag.

Table 2.3, Chemical Composition of BOS and EAF Slags (after Geiseler, 1996)

| Component | CaO (%) | $SiO_2$ (%) | $Al_2O_3$ (%) | MgO (%) | MnO (%) | $P_2O_5$ (%) | $Fe_{tot}$ (%) | $CaO_{free}$ (%) |
|---|---|---|---|---|---|---|---|---|
| BOS slag with low MgO content | 45-55 | 12-18 | < 3 | < 3 | < 5 | < 2 | 14-20 | < 10 |
| BOS slag with high MgO content | 42-50 | 12-15 | < 3 | 5-8 | < 5 | < 2 | 15-20 | < 10 |
| EAF slag with low MgO content | 30-40 | 12-17 | 4-7 | 4-8 | < 6 | < 1.5 | 18-28 | < 3 |
| EAF slag with high MgO content | 25-35 | 10-15 | 4-7 | 8-15 | < 6 | < 1.5 | 20-29 | < 3 |

Besides the use of 100% steel slag for making wearing course mixtures of BOS or EAF slags, air cooled blast furnace slag and granulated blast furnace slag and PFA achieve useful properties as a result of hydraulic binding. Work in Austria has also shown that by careful selection of slag, it is possible to use it as an aggregate in concrete road construction and in concrete floors (Alexandre et al, 1993; Sommer 1988). However steel slag has not been extensively utilised. As a result of studies into the feasibility of using steel slag in the construction industry the European Waste Catalogue (EWC, 1993) does not contain any slag products and the council of the OECD decided in 1995 (OECD, 1995) to exclude all slag products which have been specifically produced to meet both national requirements and standards from the green list of waste since they are products.

### 2.5.5 ICON Sand

ICON sand is a bi-product of Titanium Dioxide production and a summary of the production process is shown below.

#### 2.5.5.1 Chlorination

In the chlorination stage of the process, titanium oxide ores are converted to pure titanium tetrachloride. This is achieved by reacting the ores with chlorine in the chlorination reactor, then cooling the resulting gas to precipitate various metal chloride impurities, followed by further scrubbing of the gas with liquid titanium tetrachloride and condensing the titanium tetrachloride out of the product stream.

#### 2.5.5.2 Oxidation

In the oxidation stage oxygen and vaporised titanium tetrachloride are fed into an electric plasma arc reactor. The reactor product, a mixture of chlorine, oxygen and nitrogen gases with titanium dioxide powder feeds directly from the reactor into the cooling and separation section. Cooling takes place in water-cooled serpentine pipes which are scoured with sand to prevent product build-up occurring. The cooled product is fed to a filter which separates the product and sand from the tail gas which is recycled to the chlorination section. The raw product is mixed with

water to form a slurry before passing to a classifier where the **sand residues** are separated and fall down a separate chute for disposal.

#### 2.5.5.3 Raw Materials

The sand brought in to scour the serpentine pipes has a specific particle distribution so that it is small enough to be fed into the system but large enough to be easily separated in the classifier. The sand residues are wet (from slurrying) and do contain some chunks of titanium dioxide (mainly deposits scour from the walls of the serpentine cooler).

Sand residues are disposed of rather than recycled because some of the sand is ground down during scouring producing some finer particle sand. The cost of sieving and drying the sand to recover a usable fraction is not currently a cost effective process.

### 2.5.6 Lime

Although several forms of lime exist, generally it is only quicklime (calcium oxide) and hydrated lime (calcium hydroxide) that are used as binders. Quicklime, which exists either in granular or powder form, is produced from heating chalk or limestone. Hydrated lime, which is generally available as a fine, dry powder, is produced as a result of the reaction of quicklime with water. In dolomitic lime, magnesium replaces some calcium, and grey (hydraulic) lime produced from impure forms of calcium carbonate may contain some clay (Sherwood, 1993). The materials generally treated using these limes are soils (ranging from clayey gravels through to clays) and some industrial byproducts such as pulverised fuel ash (Buxton Lime, 1990).

Due to the difficulty in controlling pH when mixed with soils, lime is generally used with other reagents such as cement, PFA and carbonate ions; and additives such as hydrophobing agents, surfactants or silicates are used to improve properties and reduce permeability (Conner, 1990).

In the case of lime/clay processes the addition of lime to clay/clayey soils initiates several reactions which alter the physico-chemical properties of these soils bringing about both immediate and long term changes. When quicklime is added it initially reacts exothermically with the water to give hydrated lime. The dehydration of the system by reaction, and by steam generation, can result in benefits purely as a result of de-watering. Further, a decrease in the plasticity of clay is also associated with this reaction, and is caused by the flocculation of clay particles (Glendinning et al, 1998). This immediate modification occurs as a result of cation exchange of calcium ions for existing cations such as hydrogen and sodium ions on the clay minerals. The degree of cation exchange will depend on the mineralogy, soil composition and pore water chemistry (Al-Tabba and Perera, 2002).

In the longer term another reaction process occurs as a result of pozzolanic reactions, bringing about physico-chemical changes to lime-clay systems. This occurs when sufficient lime (quicklime or hydrated lime) is added to the soil. The lime added creates a high alkaline environment which promotes the dissolution of silica and alumina from the clay in the soil or in the impure hydrated lime. These dissolved components permit the formation of calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) gels by reacting with the calcium ions in the pore water, which in turn treat the contaminants in a manner similar to when using cement as a binder.

### 2.5.7 Portland Cement

The production of portland cement involves the firing of calcarious and argillaceous materials (usually limestone and clay, an alumino silicate).The solid raw materials are crushed and mixed in ball mills and then heated in a kiln to about 1500°C. This results in the formation of a clinker which consists of a number of compounds which set or harden when the clinker is ground into a fine powder and mixed with water.

Up until the 19th century cement was produced in vertical bottle shaft kilns. These kilns were manually charged and controlled and the irregular operation often resulted in the production of cement clinker with unpredictable and inferior properties. The capacity of the kilns was also limited to less than 300 tonnes per day (Kohlhaas, 1983). The rotary kiln was patented in 1855 by Frederick Ransome (Peray, 1986) and is the type most comonly used in the UK. The rotary kiln is essentially a large refactory lined steel tube inclined at about 3 to 5 degrees to the horizontal. At the lower end of the tube is a burner and the raw materials are fed into the higher end. The kiln is rotated, and as the materials progress down they are steadily turned into cement clinker.

## 2.6 Cement Chemistry

Portland cement is a heterogeneous mixture of four main components, see Table 2.4.

Table 2.4, Portland Cement Composition.

| Percentage | Component | Cement Chemistry Abbreviation |
|---|---|---|
| 50-70% | tricalcium silicate | $C_3S$ (alite) |
| 20 – 30% | dicalcium silicate | B-$C_2S$ (belite) |
| 5 – 12 % | tricalcium aluminate | $C_3A$ |
| 5 – 12% | calcium aluminoferrite | $C_4AF$ |

Additional components such as gypsum are sometimes added to delay the initial setting time by about 1-2 hours to ensure a period of plasticity. The hydration of dry clinker (cement) leads to the formation of:

20-25% of $Ca(OH)_2$ (portlandite, CH);
60-70% of 3 $CaO.2SiO_2.3H_2O$ (calcium silicate hydrate C-S-H); and
5-15% of all other solid phases

The principal hydration product, C-S-H, has a variable composition and its morphology is dependent on the initial Ca/Si ratio, setting conditions and

water/solid ratio. The hydration and subsequent setting of cement paste progresses through a series of competing chemical reactions (Cocke, 1990)

These reactions are exothermic and the negative enthalpies of cement hydration greatly influence:

- Hydration rate
- Microstructure
- Morphology

The mechanisms of these reactions are quite complex and not fully understood. However, two models; the gel model (or osmotic) and the crystalline model, have been proposed to explain the observed phenomena associated with cement hydration and subsequent setting.

### 2.6.1 The gel model

In this model a membrane of C-S-H gel is formed on the cement particle surface upon hydration. This membrane which is formed around the cement grains permits the inward flow of water molecules and outward migration of mainly $Ca^{2+}$ and silicate ions due to the difference of osmotic potential on both sides of the membrane. As a result, an excess of portlandite ($Ca(OH)_2$) will accumulate on the fluid side of the membrane, and precipitate. At the same time an excess of silicate ions, established on the grain side of the membrane, will produce an osmotic pressure differential which will cause the membrane to rupture periodically and then reform by extruding concentrated silicate solution.

### 2.6.2 The crystal model

The crystal model assumes that upon mixing cement with water, calcium silicate minerals dissociate into charged silicate and calcium ions. These charged silicate ions then concentrate as a thin layer on the surface of cement grains to prevent the interaction of the cement surface with water. This retards the release of calcium and

silicate ions from the cement into water. The initial hydration is followed by nucleation and growth of hexagonal crystals of calcium hydroxide that fill up the spaces and cavities between the cement grains. Meanwhile, particles of C-S-H precipitate out of water onto the silicate rich layer on the cement grains and gradually form needles or spines. Eventually needles from different cement grains come into contact with each other to form sheets of tobermorite ($Ca_5Si_6.\ O_{16}(OH)_2\ .4H_2O$).

### 2.6.3 Pozzolans

If a material is able to react with calcium hydroxide it is said to have pozzolanic activity and is called a pozzolan. The pozzolanic reaction is:

CH + S + H > C–S–H

(Mindess and Young, 2002)

Pozzolanic materials contain amorphous silica which is reactive enough to combine with calcium hydroxide to form C-S–H.

When pozzolans are mixed with Portland cement they react with the calcium hydroxide formed during hydration, the effect being to increase the proportion of C-S-H in the hydrated paste at the expense of calcium hydroxide.

Pozzolans can have a quite variable composition. Frequently, reactive alumina is also present and it can react analogously to silica.

CH + A + H > C-A-H (calcium aluminate hydrate)

Calcium aluminate hydrates can react expansively to form ettringite. Thus if a pozzolan is used to improve sulphate resistance, a material low in alumina should be used.

Pozzolanicity is the capacity of certain materials to enter into reaction with CaO or $Ca(OH)_2$ in the presence of water at room temperature to form solid and water insoluble massess.

The addition of 20-30% fly ash to Portland clinker has no practical influence on its hydration rate, especially in the first stage of reaction with water. The reaction begins with the solution of cement sulphates, since the rate of solution of anhydrite and hemihydrite in fly ashes is very slow.

Pozzolanic activity is evident from 14 days onwards, especially in the 14 to 150 day period. After 120 days, fly ash particles are practically disintegrated as a result of attack by the $Ca(OH)_2$ produced by the hydration of Portland cement. The glass phase of fly ash grains is especially affected by this attack.

### 2.6.4 Sulphate Attack

Damage can occur to cement and concrete due to chemical or physical reactions taking place within the cement. One of the most common causes for the deterioration of concrete is sulphate attack.

The major cause of sulphate attack is the reaction of gypsum with hydrated compounds in the set cement to form ettringite which results in expansion and cracking of the set concrete (Cohen and Bentur, 1988). Concretes which have been severely attacked exhibit significant cracking and spalling, concretes which have only been mildly attacked are whitish in appearance.

### 2.6.4.1 Reactions

Some of the chemical reactions which lead to formation of etteringite are shown below (S denotes sulphate, otherwise standard cement chemistry abbreviations are used):-

$3C\underline{S}H_2 + C_3A + 26H \rightarrow C_6A\underline{S}_3H_{32}$

$2C\underline{S}H_2 + C_4A\underline{S}H_{12} + 16H \rightarrow C_6A\underline{S}_3H_{32}$

$3C\underline{S}H + C_4AH_{13} + 14H \rightarrow C_6A\underline{S}_3H_{32} + CH$

Sodium and magnesium sulphates ($N\underline{S}$, $M\underline{S}$) can cause sulphate attack because they can initially react with calcium hydroxide (CH) (which is present in the set cement formed by the hydration of $C_3S$ and $C_2S$ reactions).

$N\underline{S} + CH + 2H \rightarrow C\underline{S}H_2 + NH$

$M\underline{S} + CH + 2H \rightarrow C\underline{S}H_2 + MH$

Where $N = Na_2O$ and $M = MgO$

Potasium sulphate behaves in a similar manner to sodium sulphate. The gypsum formed by these reactions then reacts with hydrated compounds to form etteringite as before. The attack by magnesium sulphate is particularly damaging because, as well as forming sparingly soluble magnesium hydroxide which forces the above reaction to form the gypsum, $M\underline{S}$ will also react with the CSH gels present in the set cement to form gypsum (Cohen and Bentur, 1988).

$xM\underline{S} + CxSyHz + (3x + 0.5y - z)H \rightarrow CSH_2 + xMH + 0.5y\underline{S}_2H$

The gypsum formed in this reaction will also react with the calcium aluminates. As well as this, the magnesium hydroxide produced in the reaction with the C-S-H gels, together with that produced by the reaction of magnesium sulphate with calcium hydroxide, can combine with silica hydrate ($S_2H$), (which is produced by the reaction with the cementitious gels) to form a non-cementitious product ($M_4\underline{S}H_{8.5}$) (Cohen and Bentur, 1988).

## 2.7 Recent Work on Synthetic Red Gypsum

Several previous studies have been carried out on the use of co-product Red Gypsum as an engineering material, these were:-

Grant, 1997, Investigation of Synthetic Red Gypsum as a Geotechnical Engineering Material, MPhil Thesis, University of Strathclyde.

Anon 2001, Use of Waste Gypsum as a Construction Material, Interpretative Report, Department of Civil Engineering, University of Newcastle upon Tyne.

Simpson 2001, Stabilisation of Peat using Co-Product Red Gypsum with GGBS, MSc Dissertation, Department of Civil Engineering, University of Newcastle upon Tyne.

Each of these reports are reviewed briefly here in chronological order.

### 2.7.1 Grant, 1997

The objective of this study was to assess the viability and possible applications of co-product Gypsum as an engineering material, in particular, to characterise the properties of Synthetic Red and White Gypsum, and to identify possible co-product disposal options and possible engineering applications of Synthetic Red Gypsum.

Grant conducted a laboratory testing programme including pH tests, moisture content tests, particle density tests, particle size distribution tests (PSD), Atterberg limit tests, linear shrinkage and free swell tests, and erodability tests to classify the geotechnical characteristics of Red and White Gypsum. Other tests conducted were compaction tests, consolidation tests, permeability and undrained shear strength tests.

Grant concluded that synthetic Red Gypsum is closely related to natural gypsum in its physical characteristics. The iron content of the Red Gypsum is erodable in still water, resulting in an undesirable loss in bond and stability.

Red Gypsum can be classified as an erodable material which is non-plastic in nature, exhibiting brittle failure on compression. The co-product displays the consolidation, permeability and shear strength characteristics of a stiff/cemented soil or soft evaporite rock (Grant 1997).

It was found that a 40% addition of clay to Red Gypsum promoted a modified co-product with desirable engineering properties. Conversely unstable clays could be stabilised by the addition of Red Gypsum. Plasticity is reduced, permeability increased, settlement reduced and shear strength increased. This is similar behaviour to that observed in lime stabilised clays.

Synthetic Red Gypsum can be characterised as an admixture for the chemical stabilisation of problematic clay soils, although further research is required to realise this potential.

### 2.7.2 Anon, 2001

The aim of the study by Newcastle University was to examine three opportunities for the engineering applications of Red Gypsum.

- As an additive to natural soil to improve the engineering properties of that soil
- As an additive to waste product producing mutually beneficial engineering properties
- As an engineering material in its own right.

In order to achieve this it was necessary to:-

- Determine the effect on soil strength, stiffness, and plasticity
- Determine the effect of soil type on material behaviour

- Determine the effect of substituting PFA and GGBS for soil
- Examine the potential for long term swell due to sulphate reduction
- Investigate solubility/erodability

Testing was conducted on a Kaolin/Bentonite mixture, London Clay, Glacial Till, PFA and GGBS, mixed with Red Gypsum at two percentage additions (20% and 60%), as well as testing 100% Red Gypsum.

Testing consisted of Compaction (BS 1377, 4.5kg method), Quick Undrained Triaxial Compression (38mm samples), 1-D Consolidation, Atterberg Limits and pinhole tests all conducted to British Standard, BS 1377 1990.

The report concluded that the addition of Gypsum to clay soils had little or no effect on the shear strength and reduced the density of the soils. There was little benefit in the addition of Gypsum to high quality clay fill except to reduce the swelling potential. The addition of Gypsum to PFA and GGBS significantly improved the shear strength whilst maintaining low density. The dispersive nature of clay soils appeared to be reduced by the addition of Gypsum, which may be due to a reduction in permeability. The addition of Gypsum was found to reduce the permeability of PFA and GGBS to values similar to that of clay soils. The addition of 60% hemi-hydrate to Glacial Till and London Clay reduced their tendency to swell on unloading.

### 2.7.3 Simpson, 2001

The research conducted by Simpson in 2001 investigated the potential for using co-product Red Gypsum mixed with GGBS as an admixture (binder) to stabilise peat. Samples of peat mixed in the laboratory with GGBS – gypsum binder at their natural moisture content, were allowed to cure at 20 C for 7, 28 and 56 days. For comparison similar samples were prepared using OPC.

Simpson carried out undrained triaxial tests, pH tests and X-ray diffraction tests on the different mixes of samples. She found that the modified soil had greater shear

strength than the original peat and that increasing the amount of binder increased the shear strength of the samples, but there was no increase in strength with curing time. This implied that pozzolanic (cementitious) reactions had not taken place. Soaking of the samples prior to testing was found to reduce the measured strength by approximately 25%.

When comparing the shear strength of peat mixed with a GGBS – gypsum binder with OPC Simpson found that the OPC mix was, on average, 65% stronger and suffered less strength reduction when soaked for 24 hours.

When testing the pH of the samples, Simpson found that the natural peat delivered to the laboratory had a pH value of 1.76. This is surprisingly low, as natural peat in the field commonly has a value of around 6.75. The addition of binder material increased the pH of the peat, and larger quantities of binder produced greater increases in pH. However, pH values rarely exceeded 8, which is significantly below the pH required for pozzolanic reactions to take place.

The X-ray diffraction testing indicated the presence of the minerals gypsum and pyrite. No evidence of ettringite or thaumasite was found. The presence of these minerals in the samples can lead to swell and the deterioration of the stabilised soil. There was no obvious difference between the mineral composition of the untreated samples and the samples treated and cured for different lengths of time indicating that curing time had no effect.

Simpson concluded that using a binder of GGBS and co-product Gypsum improved the strength and durability characteristics of the peat. However, this improvement did not increase with time. The low pH of the stabilised peat did not provide the conditions for pozzolanic (cementitious) reactions to take place. The most probable reason for the low pH values was oxidation of the peat after its extraction from the ground. With no pozzolanic reactions, no ettringite or thaumasite could form.

## 2.8 Conclusions

### 2.8.1 Cements and concretes

As covered in Section 2.2.1 there has been a long history of wastes being added to Portland Cements to modify the behaviour of cement pastes. These materials include:

- Pulverised Fuel Ash
- Silica Fume
- Ground Granulated Blast Furnace Slag

A large amount of testing and quality control programmes have been used to assess and control the properties of cements and concretes, and so as additives they are well understood to the extent that there are standards advising upon their use. Waste gyspum has previously been used to create calcium sulpho-aluminate cements. However, Beretka et al (1996), noted that given the huge volumes of waste gypsum produced anually, they are highly under utilised. However, there is a drawback that waste gypsum requires calcining at 1200°C.

In addition to PFA, various other ashes have been trialed as cement replacements. These include bottom ash from coal fired power stations, incinerated municipal waste ash, incinerated hospital waste ash, and rice husk ash. These ashes have been shown to be suitable as Portland Cement replacements in mass concretes. However, the strengths achieved by samples made with ashes have rarely achieved high strengths compared with the control samples made with Portland Cements. In many cases the use of these ashes in concretes would really be a method of disposal, rather than utilising them as an additive. Further development would be needed before these could be more widely used, and thus begin to account for the millions of tonnes produced every year.

### 2.8.2 Aggregates

The use of wastes as aggregates has also been investigated with assorted materials being considered. Recycled building rubble has been one of the most utilised wastes in concretes. Other materials trialled have been crushed glass, PET bottles, tyre chips and fibres and automotive shedder residue.

### 2.8.3 Ground Improvement

Tyres have also been used to stiffen loose granular soils. However, the vast majority of wastes used in ground improvement have been dry powders used in soil mixing. The wastes used most frequently have been materials that are also used as cement replacements in concretes such as PFA and GGBS. Although other ashes and waste gypsum have also been used.

### 2.8.4 Ground Remediation

In ground improvement many materials have been used to sorb contaminants from soils. These have included granulated tyres, wood shavings, straw, dried sewage sludge, leaf tea and tree leaves.

Shah et al (2002) also conducted an interesting study using Fly Ash as a componant of a cementitious binder used to stabilise an area of petrochemical contaminated soil. Abandoned mine workings have also been stabilised using cementitious grouts containing PFA.

### 2.8.4 Recommendations for Study

In conclusion the construction industry has a good track record of utilising waste materials, particularly in the fields of cement replacement and ground improvement. The ongoing trials of new wastes show that there is plenty of opportunity for new wastes to be exploited.

Recent studies conducted on synthetic Red Gypsum have shown that there is a potential for the material to be used as a cementitious binder. This would have a

double benefit , first by removing the need to dispose of Red Gypsum in landfill sites which has considerable financial and environmental impact, and second by replacing conventional Portland cement, the production of which has a considerable negative environmental impact (quarrying, energy costs etc.).

The review of available literature suggestes that it would be desirable to develop a Portland cement replacement utilizing Red Gypsum and other bulk wastes. Such a binder would then need to be tested with a number of engineering soils both in the laboratory and in the field in addition to conventional concrete mixes to assesss its potential as a cement replacment in mass concrete. Given the nature of Red Gypsum it would be prudent to conduct a thorough mineralogical investigation to assess the potential for etteringite formation and asertain which, if any, of the common hydrated cement minerals are formed in the hydrated Red Gypsum based cement.

# Chapter 3

# Methodology

# 3. Methodology

## 3.1 Introduction

This chapter details the methods of testing employed in the laboratory phases of the study (binder development, laboratory soil mixing, and concrete mixing). The methodology employed for the field trial is detailed in chapter 7.

### 3.1.1 Testing aims and objectives

The aims of the testing programme were

1) From the materials researched in the literature review identify which material will achieve the highest strengths and stiffnesses when mixed with Red Gypsum and therefore have potential to be used as soil mixing binders.

2) Find the optimum ratio to mix the material with Red Gypsum and the optimum water content that achieves the highest strengths and stiffnesses when tested for Unconfined Compressive Strength (UCS).

3) Assess the potential of the binder for use in civil engineering applications.

The objectives

1) Develop an optimised binder by mixing waste materials in different ratios at a range of water contents.

2) Mix the selected binder with a range of engineering soils and test its performance in terms of strength, stiffness and durability using Portland cement as a control over a range of curing periods.

3) Conduct mineralogical analysis on selected samples to identify minerals formed during curing.

4) Mix the selected binder with standard building sand and aggregate to make "gypsumcrete" and to test its performance in terms of strength, appearance and durability using concrete made with traditional Portland cement as a control.

For ease of explanation the laboratory programme presented in this chapter is split in to 3 general phases.

- Binder selection and design (aims 1&2, objective 1)
- Soil Mixing (objectives 2&3)
- Blockwork Trials (objective 4)

Each of these will be described in detail throughout this chapter

### 3.1.2 Materials used in the laboratory testing

The following is a list of the materials used in the laboratory study including the names and addresses of the suppliers.

Where portland cement is referred to the material used is Mastercrete Original (Complies to EN 1971) produced by Blue Circle, Lafarge Cements, Manor Court, Chilton OX11 ORN, UK.

The PFA used is Part 1 Pulverised Fuel Ash produced by ScotAsh Ltd, Longannet Power Station, Kincardine, FK10 4AA, UK.

The Blastfurnace Slag is Frodingham GGBS (complies BS 6699) supplied by Frodingham Cement Ltd, Brigg Road, Scunthorpe, DN16 1AW, UK.

The Lime is Calcium Hydroxide $Ca(OH)_2$ supplied by BDH Laboratory Supplies, Poole, Dorset, BH15 1TD, UK.

The China Clay is Imery Polywhite Grade E, supplied by English China Clay International, John Keay House, St Austell, Cornwall, PL25 4DU, UK.

The Fine Sand is silica sand supplied by WBB Minerals, Brookside Hall, Sandbach Cheshire, CW11 4TF, UK.

The Steel Slag is steel slag fines supplied by Appleby Group Ltd, Brigg Road, Scunthorpe. North Lincolnshire, DN16 1AW, UK.

The building sand is yellow building sand supplied by JT Dove Ltd, Orchard Street, Newcastle Upon Tyne, NE1 3NB, UK. The 10mm pea gravel is also supplied by JT Dove.

### 3.2. Binder mixing / selection

#### 3.2.1 Introduction

Previous work (Grant, 2000, Anon, 2001) has shown that when combined with PFA, OPC and most particularly GGBS the strength and stiffness of Red Gypsum is significantly increased indicating that it has a potential application as a soil binder. Research was therefore focussed on mixing Red Gypsum with these and other materials in various proportions at different water content increments. The materials the Red Gypsum was mixed with were.

- Steel Slag
- Ground Granulated Blast Furnace Slag
- Pulverised Fuel Ash

The samples were assessed for unconfined compressive strength. The results from these experiments were then used to decide what binders to use in the soil mixing phase of the programme. The binder mixing testing programme comprised of a mixing phase, curing phase and then testing, these are described in the following sections.

### 3.2.2 Mixing procedure

The first Red Gypsum mixes to be designed and tested used Red Gypsum in its original filter cake form. The water content of the filter cake was first calculated by drying in a 40°C oven so that the filter cake water could be taken into account when making samples. In all other mix trials the Red Gypsum was first dried at 40 degrees Centigrade and ground to a powder to facilitate more precise water content control.

Samples were mixed using a Hobart rotary mixing machine with a 16 litre capacity bowl. In order to obtain a homogeneous mix the Red Gypsum filter cake was placed in the mixing bowl first then the water then mixing machine started. Once a consistent paste has been made the secondary material (GGBS, PFA) was added followed by lime (2% by dry weight of the mix) to raise the pH of the mix above 10.5 so that pozzolanic reactions could take place. Once all materials were added the mixing machine was run for 10 minutes to ensure a homogenious mix. The ratios and water contents tested are detailed in Chapter 4 with the results.

Samples were prepared by compacting material using a 2.5kg rammer into a 1 litre compaction mould (see Figure 3.1) as per BS 1377, part 4 (BSI, 1990). 38mm steel sample tubes were then driven into the centre of the mould to obtain individual samples.

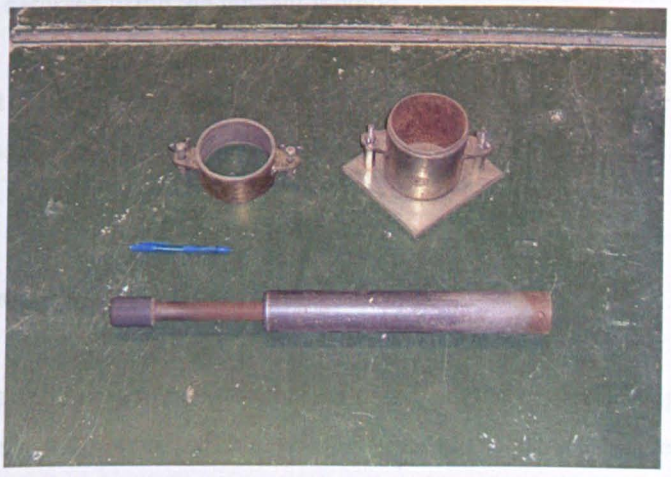


Figure 3.1 Compaction apparatus, 1 litre sample mould (upper), 2.5kg compaction rammer (lower).

As the water content of the samples was increased they became unsuitable to be compacted by the method described above, where this occurred, samples were poured

into the sample moulds and vibrated using a vibrating poker to ensure the absence of air pockets.

All samples tubes were immediately sealed with wax once samples had been taken.

### 3.2.3 Curing

Samples were cured in the steel sample cylinders for 28 days in a temperature controlled store room (20 °C, 55% relative humidity). Once the curing period was complete samples were extruded, trimmed to 76mm in length and tested immediately.

### 3.2.4 Testing

The strength of the samples was Red Gypsumed by unconfined compressive strength testing conducted according to BS 1377 part 7 (BSI, 1990). Initially the equipment used was a Shimadzu AG-250kNE test frame, the results recorded using an Autonomous Data Acquisition Unit (ADU) attached to a nearby PC. A series of technical difficulties forced a change to using an ELE Tritest 50 triaxial test frame where results were read from dial gauges (stress and strain) and recorded by hand. This had the advantage of permitting the measurement of Young's Modulus. Young's Modulus was assessed using the tangent method.

Water content of all samples was assessed by oven drying at 40° C after testing. Samples were retained for later mineralogical analysis.

## 3.3 Soil Mixing

The soil mixing phase was undertaken using data from the binder mixing (3.1) phase of the laboratory programme. After careful analysis of the data (presented in Chapter 4) from the binder testing a 50:50 Red Gypsum – GGBS binder was chosen for further research.

### 3.3.1 Soils

Four soils were chosen to mix with the selected binder. These soils were

- Glacial Till
- London Clay
- Silty sand
- Peat

These soils were selected due to their wide occurrence across the British Isles and are described in section 3.3.1.1. Geotechnical index properties are presented in Table 3.1, mineralogical data is presented in Appendix A. Particle size distribution plots are also shown in Appendix A.

Table 3.1 Soil properties, testing data is included in Appendix A

| | Glacial Till | London Clay | Silty Sand | Peat |
|---|---|---|---|---|
| **Natural Water Content (%)** | 24% | 13% | Not applicable | Not applicable |
| **Optimum Water Content (%)** | 17% | 24% | 11% | Not applicable |
| **Undrained Shear Strength (kPa)** | 58.5 kPa (at 22% water content) | 97 kPa (at 30% water content) | 31.5 kPa (at 15% water content) | 4.6 kPa (at 420% water conent) |
| **Natural pH** | 7.9 | 5.5 | 5.8 | 4.9 |
| **Degree of Humification (Von Prost)** | Not applicable | Not applicable | Not applicable | 9 |
| **Primary minerals** | Quartz Kaolinite, Illite, chlorite Mica (Barlow et al, 1997) | Quartz Mica smectite, kaolinite (Avery and Bollock, 1977) | Quartz, kaolinite | Not applicable / organic |

#### 3.3.1.1 Glacial Till

The Glacial Till was obtained from Stobswood Opencast Coal Site in North Northumberland, UK. There are many types of Glacial Till occurring in this region of the UK, the type selected for the soil mixing programme was a red till.

"red brown with very occasional black, soft to firm, very slightly sandy, slightly gravely CLAY. Gravel is fine to coarse, angular to sub-rounded and composed largely of medium grained sandstone" (BS 5930 description (BSI, 1999)).

#### 3.3.1.2 London Clay

The London Clay used in the soils mixing programme was part of a batch obtained by Newcastle University and kept in storage bins in the soils laboratory. London Clay as the name suggests occurs across the south of England around London, the material is generally an grey brown hard clay with varying amounts of silt, sand and gravel present.

"grey-white with occasional yellow, stiff to hard very slightly gravely fissured CLAY, gravel is fine and angular" (BS 5930 description (BSI, 1999)).

#### 3.3.1.3 Peat

Peat was selected as it is a particularly problematical soil in civil engineering projects in the UK and Ireland. The peat selected for this research programme was Irish Moss Peat, obtained from a local garden centre. This peat had been dried and processed for use as a garden soil conditioner.

#### 3.3.1.4 Silty Sand

Alluvial sand with silt content is a material frequently associated with difficult excavation conditions. In both dry and wet conditions the material has very low to non existent cohesion leading to side wall collapses in excavations of all sizes. This type of soil is encountered widely across the UK, particularly in low lying flood plains of river valleys, areas which are commonly developed for housing and industrial use.

In this case it was decided not use a "real" soil but instead to mix a silty SAND in the lab. The soil was made by mixing 70% fine to medium sub-angular sand with 30%

Grade E Kaolin for English China Clay ltd. Grade E china clay is predominantly silt with a small clay content.

### 3.4 Soil and Binder Mixing

It was decided to use 3 different binder concentrations, 5% 10% and 20% by dry weight. The water contents at which the samples were mixed were decided on the basis of the results of a 2.5kg Rammer Compaction Tests conducted in accordance with BS 1377 (BSI, 1990). Water contents are shown in Table 3.2.

The soils used were initially oven dried at 200° C in order to ensure accurate water content control and then (except in the case of the Peat) machine ground. The Red Gypsum used in the soil mix testing was dried at 40° C in an oven and then ground into a powder.

The binder was pre mixed (GGBS, Red Gypsum and lime), then added to the soil and dry mixed in a mixing machine (in order to ensure adequate pH would be achieved each of the soils was first mixed with 5% of the binder and then tested in accordance with BS 1377, part 3, (BSI, 1990) with the exception of the peat it was found that after a period of 8 hours sufficient pH was attained when lime made up 2% of the binder). Once a homogenous mix had been achieved the necessary volume of water was added and left in the mixing machine for 10 minutes.

Table 3.2 Target sample properties.

| | **Silty sand** | **London Clay** | **Glacial Till** | **Peat** |
|---|---|---|---|---|
| Mix water content (%) | 15 | 30 | 22 | 400 |
| Bulk sample mass (g) | 189 | 168 | 181 | 80 |
| Sample bulk density ($Mg/M^3$) | 2.1 | 1.9 | 2.0 | 1 |

Samples were prepared by measuring a pre-calculated mass of soil (see Table 3.2 for values) to achieve a consistent density, these were calculated from compaction test

data, and tamping/compressing the soil into a split sample mould of dimensions 38mm diameter, 76 mm length (see Figure 3.2). Once the soil had been tamped into the mould (in three equal layers) the mould was placed in a hydraulic press (see Figure 3.3), compacting the samples into the correct dimensions and driving out air. This method allowed the production of samples of consistent dimensions, water content and density at a relatively rapid rate. Samples were prepared in batches of 9 and sealed immediately in order to prevent samples drying during the mixing/sample forming process.

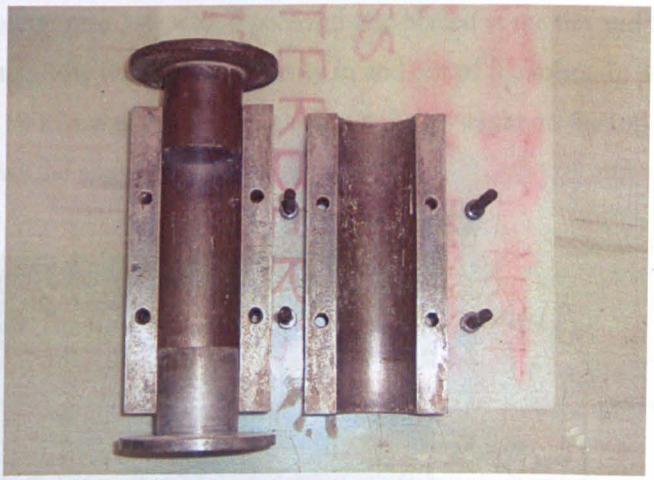


Figure 3.2 Split sample mould

Figure 3.3 Hydraulic press used to form samples.

The samples were cured in two different ways, dry and soaked. The dry cured samples were immediately placed in 38mm UPVC plastic sample tubes and the ends sealed with wax, these samples were immediately placed in a temperature controlled store room (20 °C). The philosophy behind the dry cured samples was to try and simulate soil mixing close to the ground surface above the water table in unsaturated soils with no access to more water.

The soaked cured samples were also placed in 38mm UPVC plastic sample tubes but these sample tubes were not wax sealed. Instead the sample tubes were placed in curing tanks (see Figure 3.4) with a geosynthetic placed at the top and the base of the tubes in order to allow water to flow easily in and out of the tubes. In addition to this a surcharge of 18 kPa was applied by placing a 204g weight on the top of the sample. Figure 3.4 shows the soaked curing equipment.

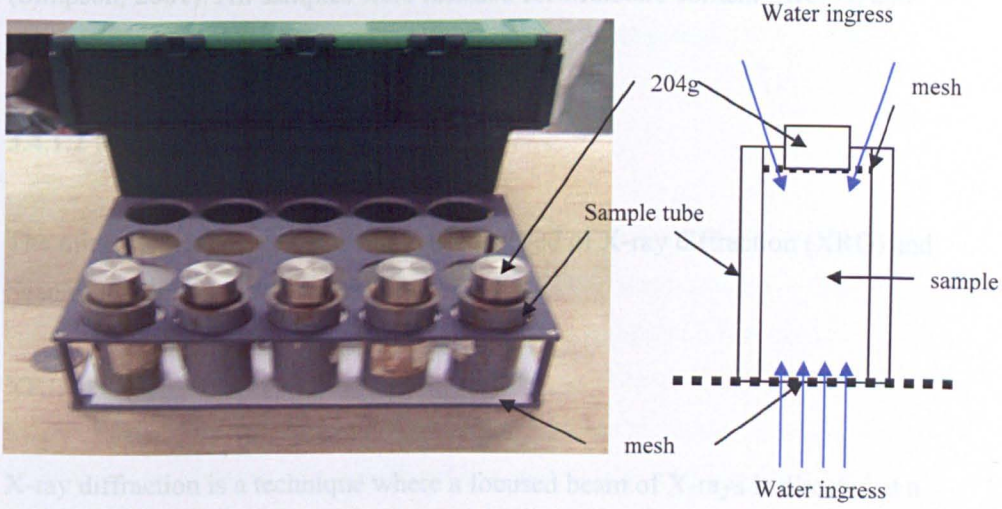



Figure 3.4 Soaked curing apparatus.

The philosophy of the soaked cured samples was to simulate soil mixes at depth under a confining pressure below the water table with access to free water (Okumura, 1980).

The curing periods chosen for the samples were 7, 14, 28, 56, 112 days. Three samples were created for each curing period.

### 3.4.1 Testing

The testing programme for the soil mixing consisted of 2 separate parts

- Shear Strength Tests
- Mineralogical Tests

#### 3.4.1.1 Shear Strength Tests

An assessment of the un-drained shear strength of the samples was made using un-drained triaxial tests, conducted in accordance with BS 1377, part 7 clause 8 (BSI, 1990) the samples were tested at a strain rate of 1.23mm/min, (1.5 %/mm) and a confining pressure of 100kPa, this was chosen as it was used in a previous study (Simpson, 2001). All samples were retained for Moisture content (MC %) and mineralogical analysis after test.

#### 3.4.1.2 Mineralogical Testing

The mineralogical analysis of samples consisted of X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis.

**XRD**

X-ray diffraction is a technique where a focused beam of X-rays is directed at a powder sample of material and the pattern of the diffracted rays is recorded, the pattern can be used to identify the mineralogy of crystal grains in the powder. The process is described by Eberhart, 1991.

The machine used for the X-ray analysis was a PANalytical X'Pert Pro diffractometer, fitted with an X'Celerator and a secondary monochromator. The X'Celerator is a relatively new attachment to the X'Pert and has the effect of giving a good quality pattern in a fraction of the time of a traditional diffractometer. The secondary monochromator eliminates fluorescent scattering from the specimens, and

so results in a better peak : background ratio for samples containing transition metals and rare earths.

The scans were conducted in the range 7-50 degrees (for binder samples) and 7-70 deg (for soil mix samples), programmed to a nominal step size of 0.033 deg 2-theta and time per step of 100 seconds. The scans were carried out in "continuous" mode, rather than "step scan" and took approximately 26 minutes per run for the 7-70 degree range. Using conventional X-ray diffraction equipment these scans would have taken 53 hours, the reason for the much swifter analysis is the use of the X'Celerator.

Radiation is Cu K-alpha: lambda = 1.54180 angstroms, or Cu K-alpha1: lambda = 1.54059 angstroms

Phase identification was carried out by means of the X'Pert accompanying software program High Score and the ICDD database, Sets 1-49 (1999).

**SEM**

SEM analysis is a technique where a sample is analysed under a beam of electrons and is therefore able to achieve a much higher level of magnification than conventional optical microscopes. For this research the soil mixed samples were prepared as polished sections. The constituent materials of the binder (GGBS, Red Gypsum etc) were analysed in powder form. For a more complete description of the SEM technique consult "Structural and chemical analysis of materials : X-ray, electron and neutron diffraction, X-ray ion spectrometry, electron microscopy" (Eberhart, 1991).

Because of the costs and time involved in XRD and SEM analysis it was not possible to test all of the samples that underwent triaxial testing. Where possible 28 day soaked samples with 20% binder concentrations were tested.

The Scanning electron microscope used in the experiments was an FEI XL30 ESEM FEG using a Centaurus backscattered electron detector to collect the images

reproduced in this thesis. Point elemental analysis was done on individual grains within the samples using a Rontec Quantax Energy Dispersive X-ray analyser.

## 3.5 Blockwork Trials

### 3.5.1 Testing aims and Philosophy

During the preliminary stages of the soil mixing phase of the research the potential to use the Red Gypsum – GGBS binder as a replacement for Portland cement in concrete mixes was identified. Because of time pressure the testing was carried out by final year undergraduate students and technicians under my supervision.

The blockwork research was done in two parts. A preliminary research project was conducted to asses the viability of using the binder in place of OPC and comparing the UCS to that of a conventional concrete mix made with OPC. This research was conducted by Ng (under supervision) and formed his final year BEng Civil Engineering project. After analysing the results from this initial phase it was decided to conduct a more comprehensive programme of testing with an aim to finding a suitable mix for use in the production of paving blocks.

The research programme was split into 2 parts, after a total of 15 suitable mixes (including controls) were selected an initial programme of workability testing was conducted in order to select suitable water contents for the next phase of research.

In the second phase of the research cubes were made from the mixes at 3 different water contents (established by previous phase) and tested for UCS at 1, 7, and 28 days with non destructive testing also being conducted using Ultrasonic Pulse Velocity testing equipment.

### 3.5.2 Materials Used

In addition to the materials listed previously the following materials were used in this programme:

- Building Sand (PSD curve included in Appendix B)
- Pea Gravel (PSD curve included in Appendix B)
- ICON sand (PSD curve included in Appendix B)
- Ochre
- Ordinary Portland Cement

Information on the characteristics of these materials is given in the Chapter 2.

### 3.5.3 Initial Trial

In the initial trial only one Red Gypsum mix was tested, comprising a 4:2:1 (aggregate, sand, Red Gypsum - GGBS binder) mix ratio. A control mix composed of the same materials at the same water content but with Portland cement substituted for the GGBS – Gypsum binder was also tested.

15cm cubes were prepared and UCS and UPV (see section 3.5.4.3) tested after curing periods of 7, 14 and 28 days, soaked in curing tanks at temp 20C. Samples were retained after UCS testing for XRD analysis.

### 3.5.4 Main Programme

As stated previously this phase of the research was split into two parts:

- Workability
- Cube Testing

### 3.5.4.1 Workability Phase

Before attempting to make cubes for the strength testing it was decided to test the intended mixes for workability in order to asses what water contents would be most appropriate for cube mixes. The mixes and controls to be tested are detailed in Chapter 6. The method chosen to asses the workability of the mixes was the slump test, conducted in accordance with BS 1881, part 102 (BSI, 1983).

The mixer used was a horizontal barrelled electric powered unit, 12 kg of each mix was prepared and the water contents increased in increments at each increment 3 slump tests were performed on the mix and a sample retained for water content checking. This process was continued until the mixes became slurries. The mixer used was a pan type, with non-motor driven mixing fins.

### 3.5.4.2 Cube Preparation

Results from the workability testing programme were used to select the water contents for the cube mixes. Samples were mixed in the same mixer as the previous phases.

Samples were compacted in 10cm cube moulds in 3 layers, samples were then sealed with plastic film and dry cured for 1,7, and 28 day curing periods.

### 3.5.4.3 Cube Strength Testing

The cubes were subjected to two main types of test Uni-axial Compressive Strength (UCS) in accordance with BS 1881, part 116 (BSI, 1983) and ultrasonic pulse velocity (UPV) p-wave BS 1881, part 203 (BSI, 1986). Tests were conducted at 1, 7, and 28 days.

Before testing sample dimensions and mass were also measured so that density could be calculated and samples were retained after UCS testing for water content check and mineralogical analysis.

# Chapter 4

# Binder Results

# 4. Binder Results

## 4.1 Physical Testing

As stated in the methodology section, Red Gypsum was mixed with GGBS, Steel Slag, and PFA with small (1%) additions of lime to boost pH values. Results from each of the different material additions will be presented separately in this section. For all samples pre and post water content, bulk and dry density, and shear strength were measured, and due to a change in equipment during the testing programme it was also possible to measure the Young's Modulus of the Steel Slag and PFA samples. X-ray diffraction and SEM analysis were conducted on some of the binder samples after physical testing of the specimens was complete. The curves plotted in the graphs in this chapter are interpolated using Microsoft Excel 2003. These curves are plotted for presentation purposes, and results should only be inferred from the points plotted on the graphs.

### 4.1.1 PFA

Red Gypsum – PFA samples were mixed at two ratios, 50:50 and 30:70, 4 samples of each ratio at water contents between 9.4 - 40.6% and 35.7 – 66.9% respectively (see Table 4.1).

#### 4.1.1.1 Density

Tables showing the full results from Red Gypsum : PFA samples are included in Appendix B. Figures 4.1 and 4.2 show the relationship between water content and bulk density and dry density respectively. Densities have been calculated for handling purposes and assess the effect of packing on strength. Bulk and dry density are as defined in BS 1377, (BSI, 1990), the temperature for water content drying was 40°C. It can be seen from Figure 4.1 that the peak bulk density of a 50:50 Red Gypsum : PFA mix is 1.88 $Mg/m^3$ at a water content of 30.2%, peak dry density 1.52 at 30.2% water content (Figure 4.2). Unfortunately the peak bulk density of the 30:70 ratio did not fall within the range of water contents tested (see Figure 4.1), the highest bulk

density measured was 1.71 $Mg/m^3$ at water content of 35.7%. The same case applied to dry density, the highest measured being 1.41 $Mg/m^3$; also at 35.7% water content.

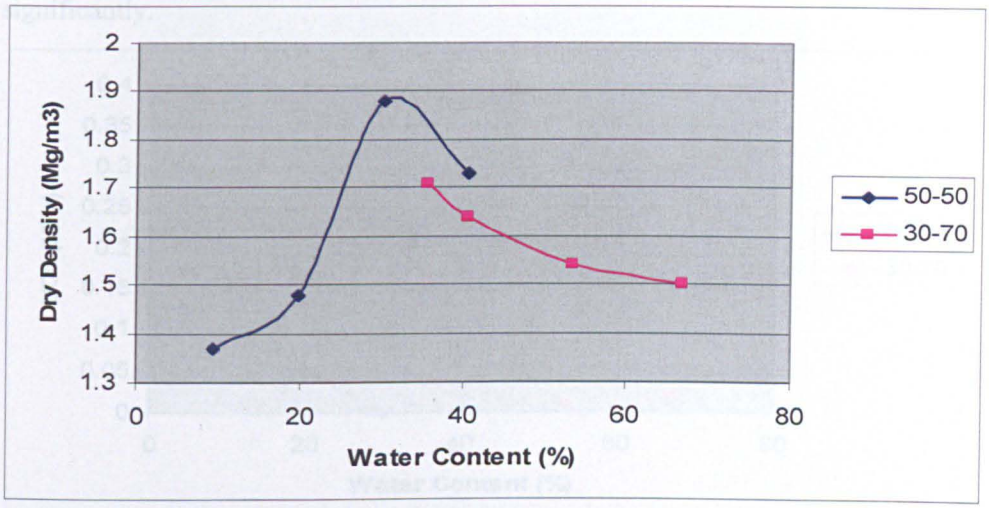



Figure 4.1 Red Gypsum – PFA bulk density results.

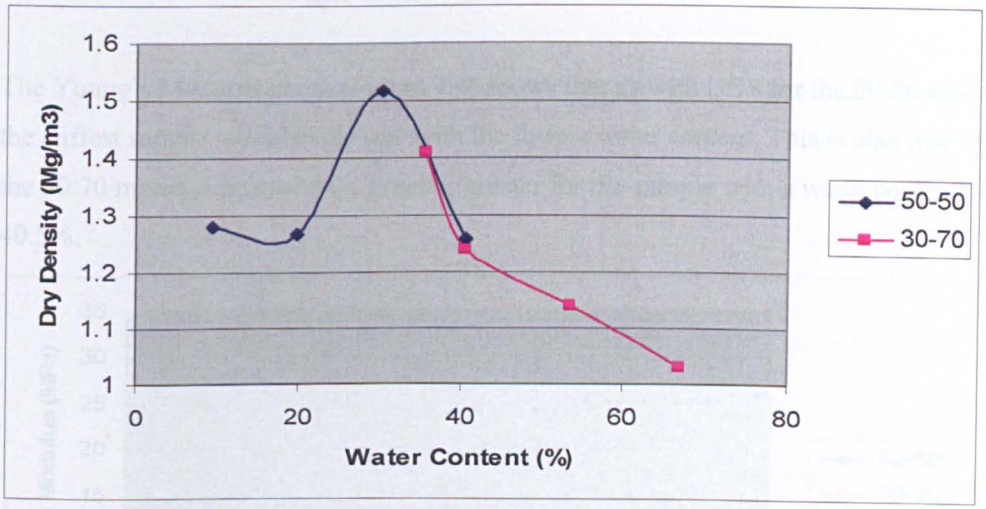



Figure 4.2 Red Gypsum – PFA dry density results.

### 4.1.1.2 Strength and Stiffness

Figures 4.3 and 4.4 show the relationship between water content and UCS in the two mix ratios tested. Figure 4.3 shows that increasing water contents from 10 to 32% reduced the UCS of 50:50 ratio samples. The sample mixed at 40.6% water content was too weak to test. The highest strength achieved was 0.06 MPa at 9.4% water content.

Results from the 30:70 mix show that the peak UCS is 0.34 MPa at 40.5% water content. Further increasing the water content caused the samples to reduce in strength significantly.

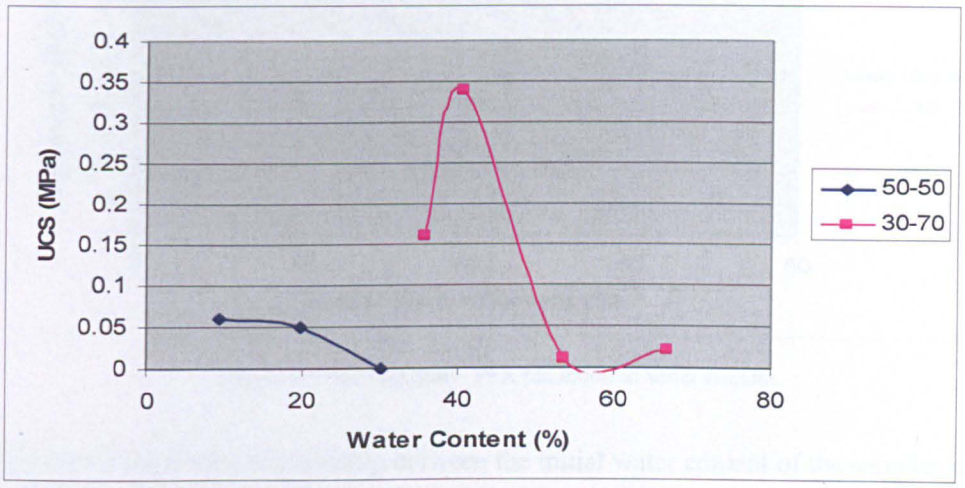



Figure 4.3 Red Gypsum – PFA UCS results

The Young's Modulus graph (Figure 4.4) shows that as with UCS for the 50:50 mix, the stiffest sample was also the one with the lowest water content. This is also true of the 30:70 mixes, despite the UCS being greater for the sample with a water content of 40.5%.

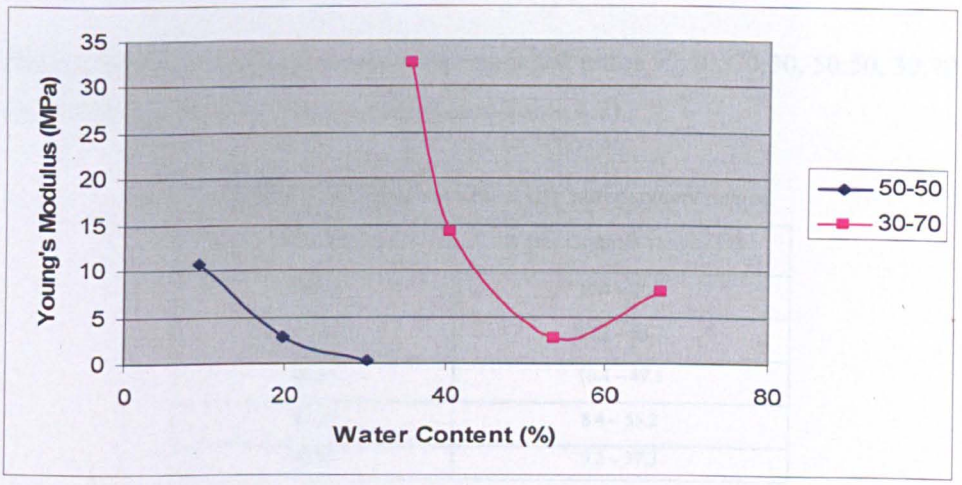



Figure 4.4 Red Gypsum – PFA Young's Modulus results.

Figures 4.3 and 4.4 also indicate that significantly higher strengths and stiffness are achieved by the 30:70 mix than the 50:50 mix.

#### 4.1.1.3 Reduction in Water Content

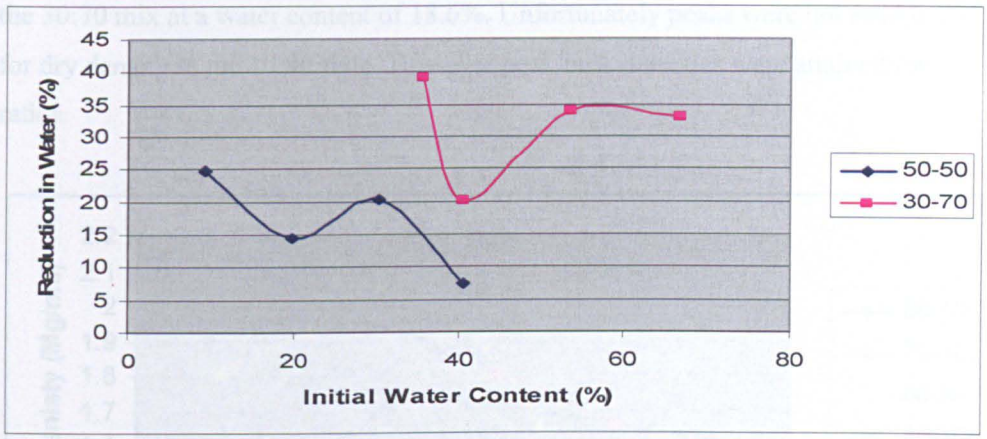



Figure 4.5 Red Gypsum – PFA reduction in water content.

Figure 4.5 shows the relationship between the initial water content of the samples and percentage of the initial water content lost during the curing time. As can be seen in Figure 4.5, in general, samples with higher initial water contents lost a lower proportion of water during curing. This has been conducted to evaluate the quantity of water used during hydration of the binder.

### 4.1.2 Steel Slag

Red-Gypsum – Steel Slag samples were mixed at ratios 90:10, 70:30, 50:50, 30:70 and 10:90 at a range of water contents (see Table 4.2).

Table 4.2, Red Gypsum - Steel slag water content ranges.

| RG : Steel Slag ratio | Water Content range (%) |
|---|---|
| 90:10 | 10.4 – 70.4 |
| 70:30 | 12.2 – 58.7 |
| 50:50 | 10.4 – 49.5 |
| 30:70 | 8.4 – 55.2 |
| 10:90 | 9.2 – 37.3 |

#### 4.1.2.2 Density

Figures 4.6 and 4.7 show plots of bulk and dry density vs water content; full tabulated results are contained in Appendix B. As can be seen from Figures 4.6 and 4.7, the

highest densities (2.09 Mg/m$^3$ bulk density and 1.88 Mg/m$^3$ dry density) occurred in the 30:70 mix at a water content of 18.6%. Unfortunately peaks were not established for dry density in the 10:90 ratio. However peak bulk densities were attained for all ratios.

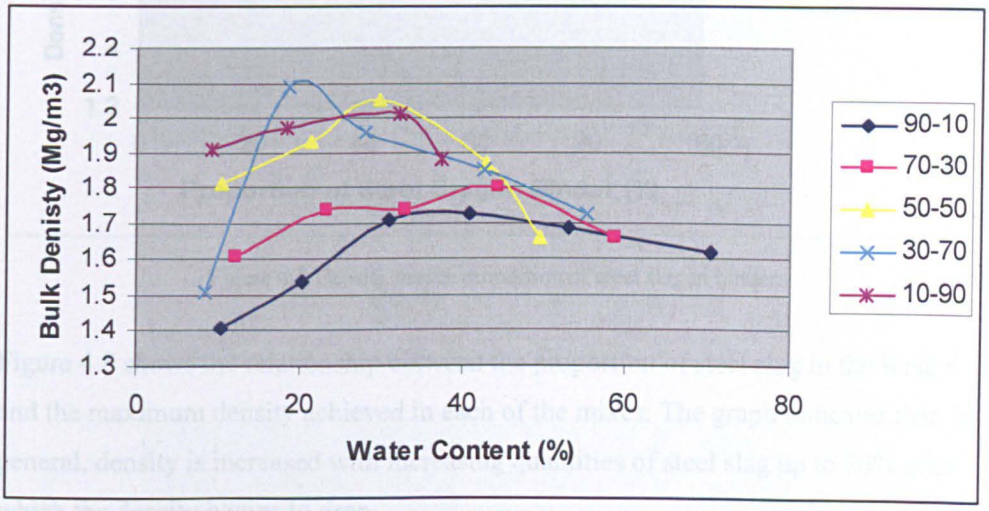



Figure 4.6 Red Gypsum - Steel Slag bulk density results.

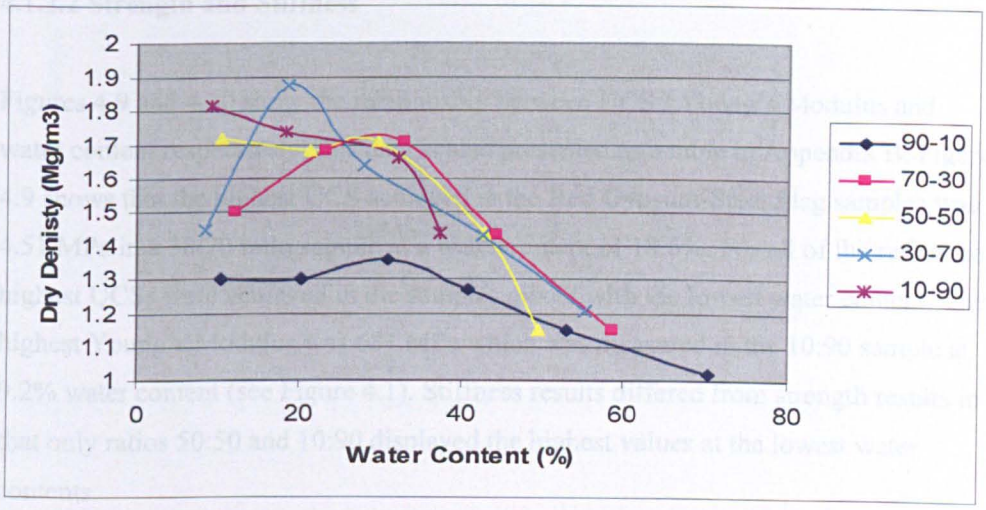



Figure 4.7 Red Gypsum – Steel Slag dry density results.

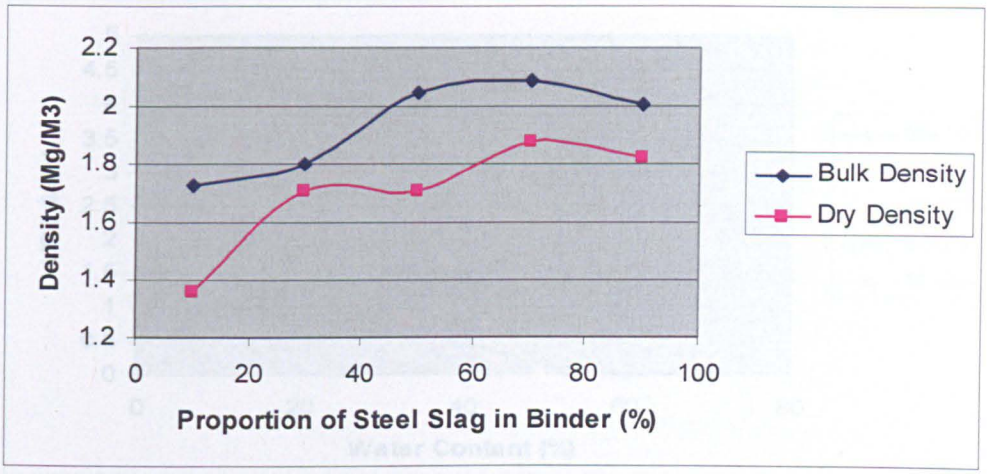



Figure 4.8 Density versus proportion of steel slag in binder.

Figure 4.8 shows the relationship between the proportion of steel slag in the binder and the maximum density achieved in each of the mixes. The graph indicates that, in general, density is increased with increasing quantities of steel slag up to 70% after which the density begins to drop.

#### 4.1.2.2 Strength and Stiffness

Figures 4.9 and 4.10 show the relationship between UCS / Young's Modulus and water content respectively; this data is also presented as a table in Appendix B. Figure 4.9 shows that the highest UCS achieved in the Red Gypsum-Steel Slag samples was 4.51 MPa in a 30:70 ratio sample at a water content of 18.6%. For all of the ratios the highest UCSs were achieved in the samples mixed with the lowest water content. The highest Young's Modulus was 681 MPa which was measured in the 10:90 sample at 9.2% water content (see Figure 4.1). Stiffness results differed from strength results in that only ratios 50:50 and 10:90 displayed the highest values at the lowest water contents.

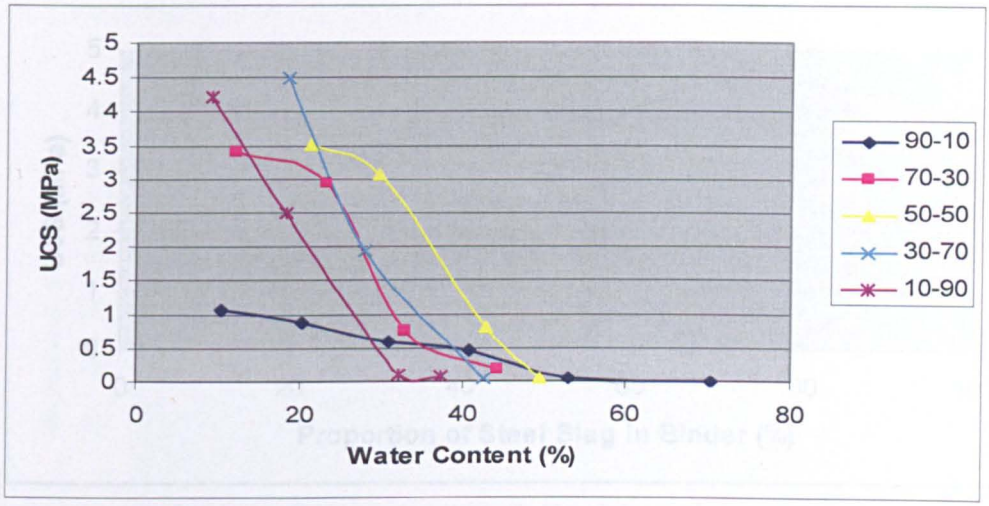



Figure 4.9, Red Gypsum – Steel Slag UCS results

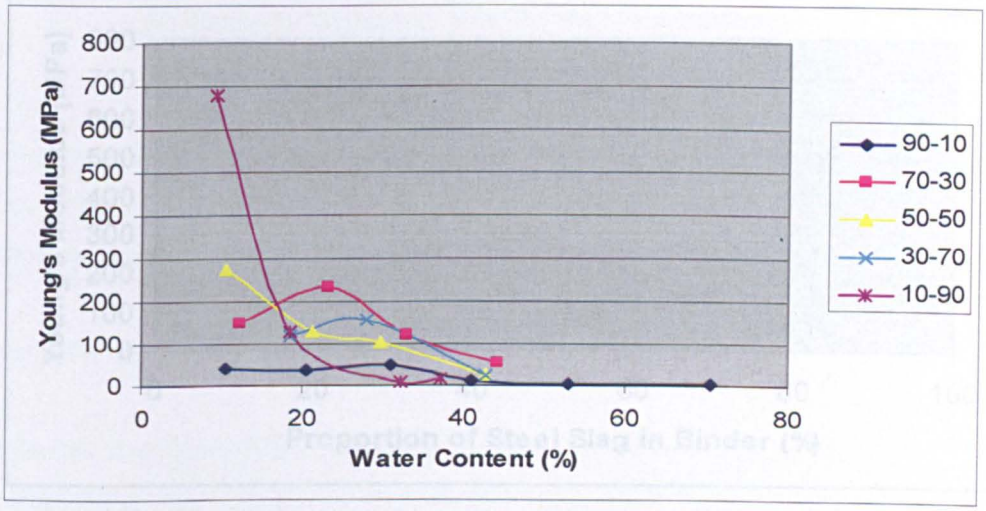



Figure 4.10, Red Gypsum – Steel Slag Young's Modulus Results

Figures 4.11 and 4.12 show the variation in Maximum UCS and Young's Modulus with increasing proportions of Steel Slag in the binder.

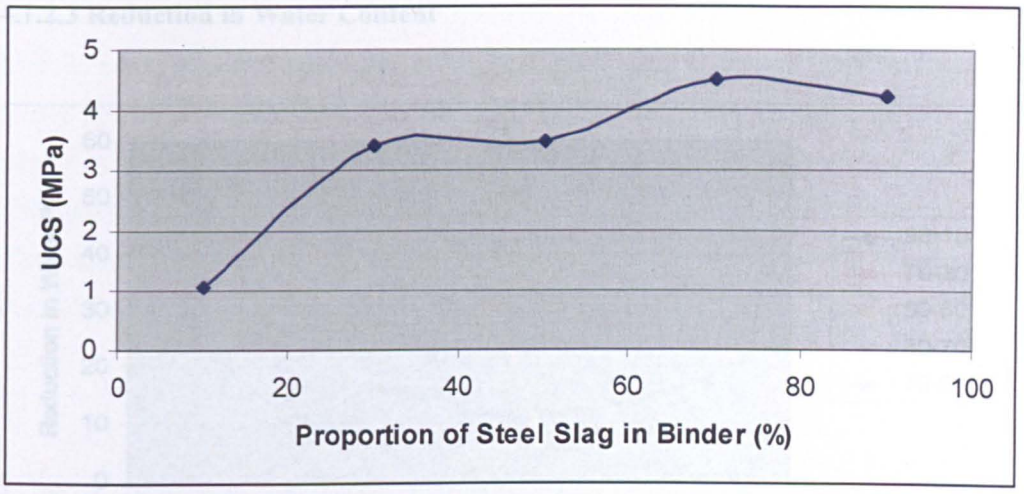



Figure 4.11, Red Gypsum – Steel Slag, maximum UCS results.

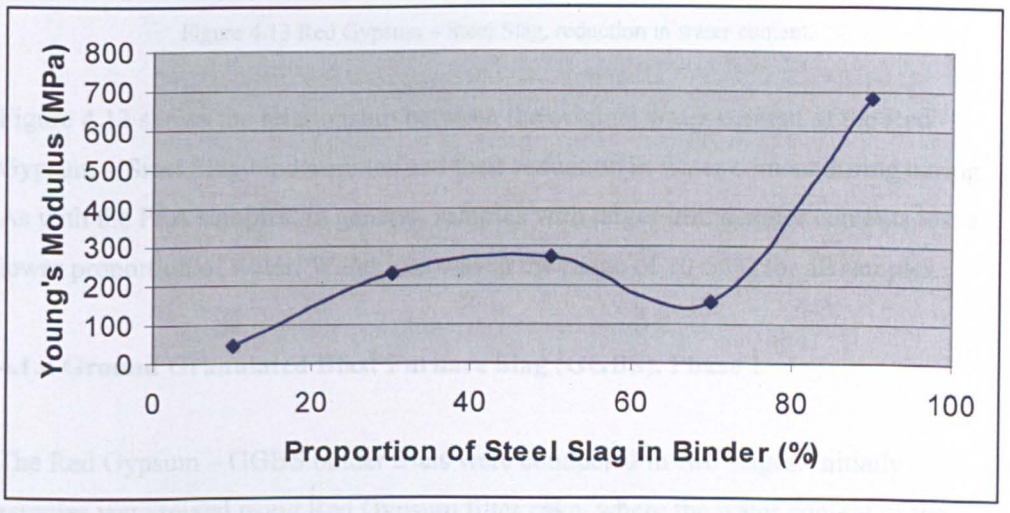



Figure 4.12, Red Gypsum – Steel Slag, maximum Young's Modulus Results

Figure 4.11 shows that strengths increase with decreasing quantity of Red Gypsum until a 30:70 ratio is reached. The strength then drops at the 10:90 ratio. Stiffness also generally increases with decreasing quantity of Red Gypsum as can be seen in Figure 4.12, with the exception of the 30-70 ratio although given the UCS results this is possibly an anomalous reading

#### 4.1.2.3 Reduction in Water Content

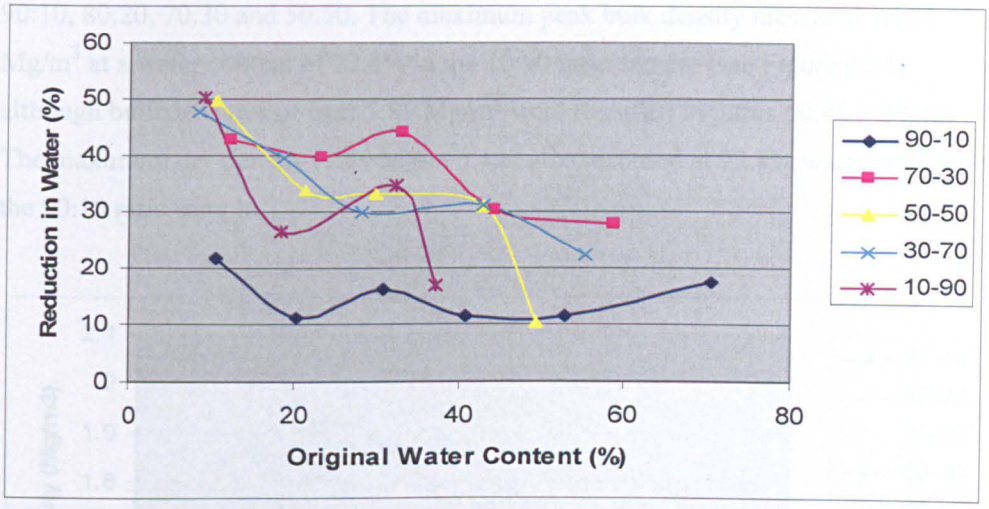



Figure 4.13 Red Gypsum – Steel Slag, reduction in water content.

Figure 4.13 shows the relationship between the original water content of the Red Gypsum – Steel Slag binder mixes and their reduction in water content during curing. As with the PFA samples, in general, samples with larger initial water contents lost a lower proportion of water. Water loss was in the range of 10-50% for all samples.

### 4.1.4 Ground Granulated Blast Furnace Slag (GGBS), Phase 1

The Red Gypsum – GGBS binder trials were conducted in two stages. Initially samples were mixed using Red Gypsum filter cake, where the water content of the fresh cake was measured and this was then taken into account when mixing the samples. It was found that water contents were difficult to control, so in the second phase the filter cake was dried at 40°C for 5 days and then pulverised before mixing allowing better control of water content. Also two test specimens of each sample were mixed.

#### 4.1.3.1 Density

Figures 4.13 and 4.14 show the relationship between water content and bulk and dry density; this data is also presented as a table in Appendix B. Unfortunately due to

using fresh Red Gypsum filter cake it was not possible to make dry mixes, therefore peak bulk and dry densities could not be measured for Red Gypsum - GGBS ratios 90:10, 80:20, 70:30 and 50:50. The maximum peak bulk density measured was 2.00 $Mg/m^3$ at a water content of 22.8% in the 10:90 ratio sample (see Figure 4.13), although bulk densities of over 1.85 $Mg/m^3$ were recorded in ratios 60:40 – 20:80. The maximum dry density (1.89 $Mg/m^3$) was also recorded at 22.8% water content in the 90:10 ratio sample.

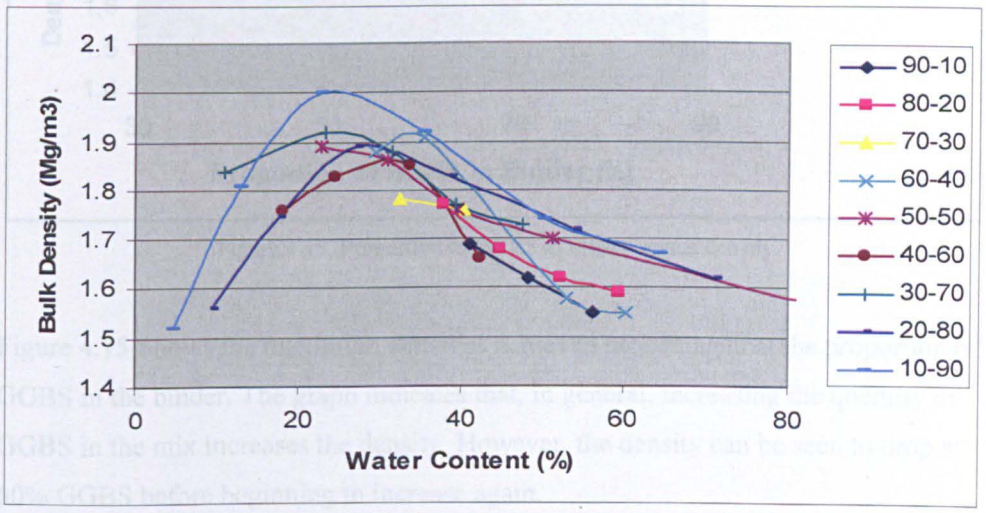



Figure 4.13, Red Gypsum filter cake – GGBS bulk density results

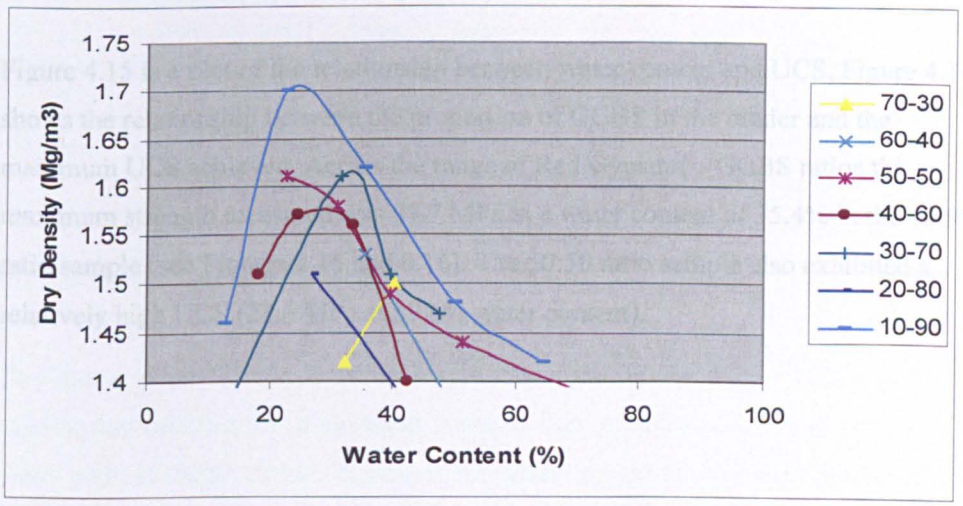
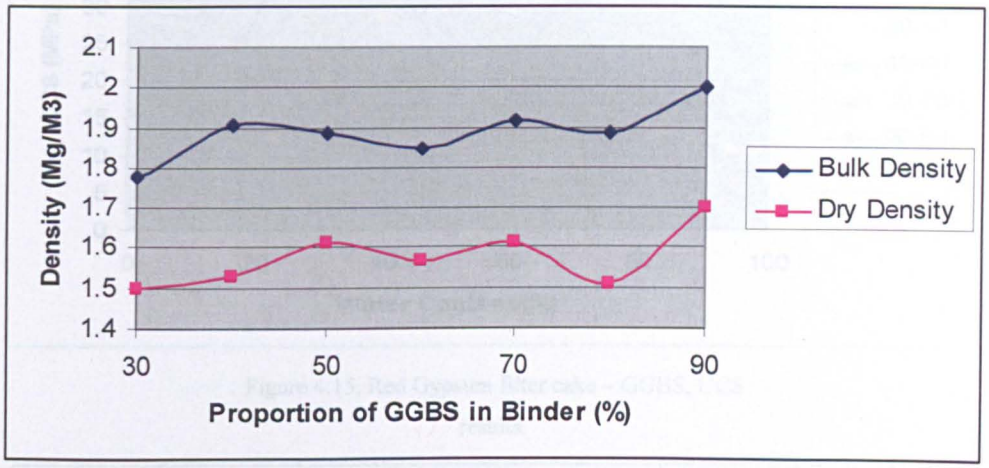



Figure 4.14, Red Gypsum filter cake – GGBS dry density

From Figures 4.13 and 4.14 it can be observed that, in general, peak densities are achieved at around 30% water content.




.Figure 4.15, Proportion of GGBS in binder versus density.

Figure 4.15 Shows the maximum densities achieved plotted against the proportion of GGBS in the binder. The graph indicates that, in general, increasing the quantity of GGBS in the mix increases the density. However, the density can be seen to drop at 60% GGBS before beginning to increase again.

#### 4.1.3.2 Strength

Figure 4.15 is a plot of the relationship between water content and UCS, Figure 4.16 shows the relationship between the proportion of GGBS in the binder and the maximum UCS achieved. Across the range of Red Gypsum – GGBS ratios the maximum strength measured was 39.7 MPa at a water content of 35.4% in the 10:90 ratio sample (see Figures 4.15 and 4.16). The 50:50 ratio sample also exhibited a relatively high UCS (28.6 MPa at 39.6% water content).

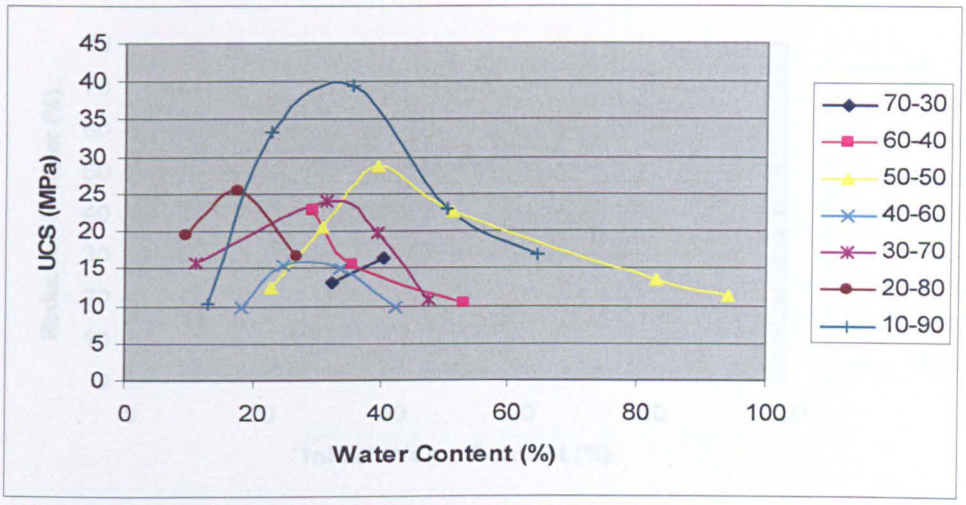



Figure 4.15, Red Gypsum filter cake – GGBS, UCS results.

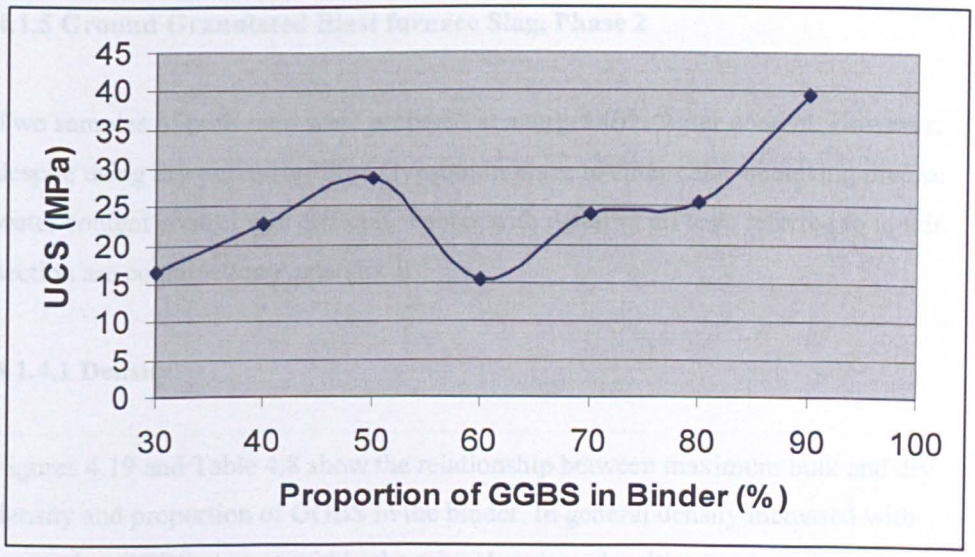



Figure 4.16, Red Gypsum filter cake - GGBS, Maximum UCS results

### 4.1.3.3 Water Content Reduction

Post curing water contents were measured after UCS testing. Figure 4.17 shows the relationship between the initial water content of the samples and the reduction in water content during curing. In general the water content of the samples reduced by 50% in the 28 day curing period but this increases with initial water content.

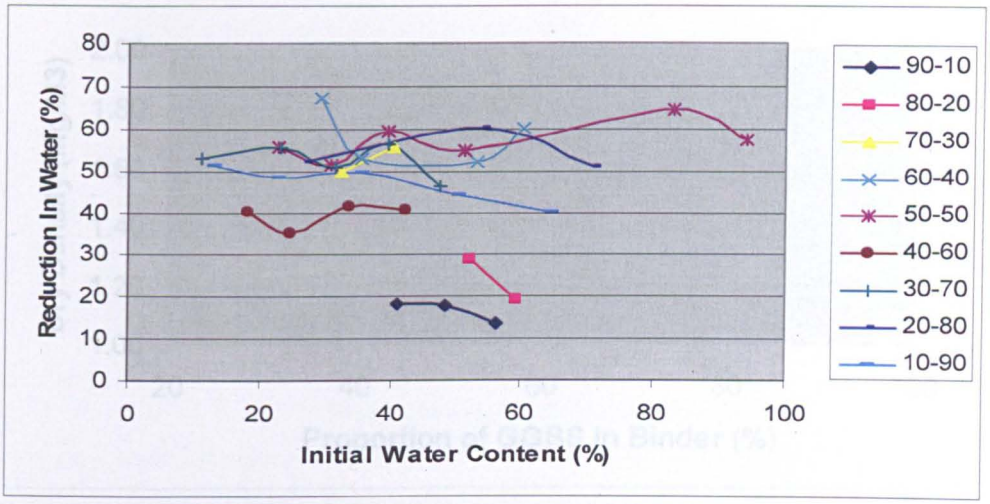



Figure 4.17, Red Gypsum filter cake – GGBS, water content reduction

### 4.1.5 Ground Granulated Blast furnace Slag, Phase 2

Two samples of each ratio were prepared at a target 40% water content. However, despite using dry pulverised Red Gypsum in place of filter cake, achieving precise water content control was difficult. Tables with detail of all tests referred to in this section are contained in Appendix B

#### 4.1.4.1 Density

Figures 4.19 and Table 4.8 show the relationship between maximum bulk and dry density and proportion of GGBS in the binder. In general density increased with increasing GGBS content within the mix. However, densities can be seen to dip at 60% GGBS before climbing again. The dry density result at 90% GGBS is an anomaly most likely caused by voids forming in the specimens.

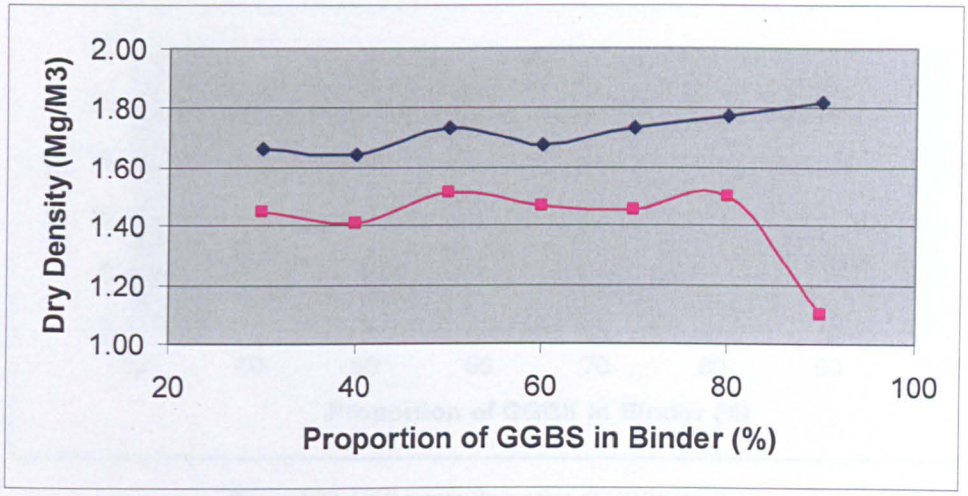



Figure 4.19, 2nd Phase Red Gypsum – GGBS density results.

### 4.1.4.2 Strength

In general the strength of the samples increased with increasing proportion of GGBS in the mix, the highest strength achieved being 23.3 MPa. Table 4.3 shows the mean strengths achieved in each of the Red Gypsum – GGBS ratios tested and their corresponding density and water content results, the table shows that there was considerable variation between samples made from the same mix.

Table 4.3, Red Gypsum – GGBS 2nd phase average results

| Red Gypsum : GGBS ratio | Water Content (%) | UCS (MPa) | Bulk Density (Mg/m3) | Dry Density (Mg/m3) | Water Content reduction (%) |
|---|---|---|---|---|---|
| 70-30 | 47.5 | 4.92 | 1.67 | 1.45 | 45.2 |
| 60-40 | 40.7 | 6.92 | 1.65 | 1.41 | 22.6 |
| 50-50 | 38.3 | 10.85 | 1.74 | 1.52 | 40.0 |
| 40-60 | 48.0 | 10.0 | 1.68 | 1.47 | 42.5 |
| 30-70 | 42.5 | 15.0 | 1.74 | 1.46 | 43.7 |
| 20-80 | 43.1 | 12.4 | 1.78 | 1.5 | 42.8 |
| 10-90 | 41.5 | 23.3 | 1.82 | 1.1 | 38.1 |

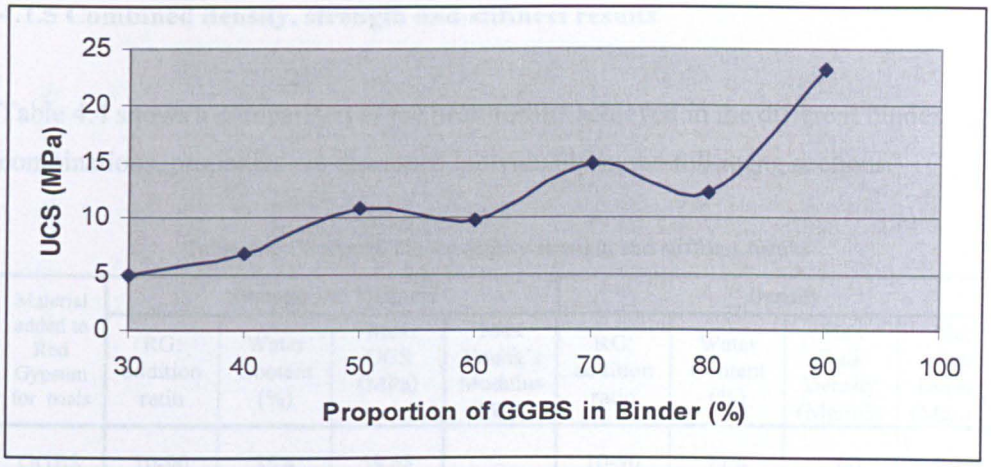



Figure 4.20, UCS versus Proportion of GGBS in binder.

Figure 4.20 shows the relationship between the proportion of GGBS in the binder and the UCS achieved. It indicates that, in general, increasing the quantity of GGBS in the binder increases the UCS.

### 4.4.2.3 Water content reduction

Figure 4.21 plots the relationship between the proportion of GGBS in the binder and the percentage water reduction during curing. Apart from mix ratio 60:40 Red Gypsum: GGBS samples, mixes exhibited reductions in water content of between 38 and 44%.

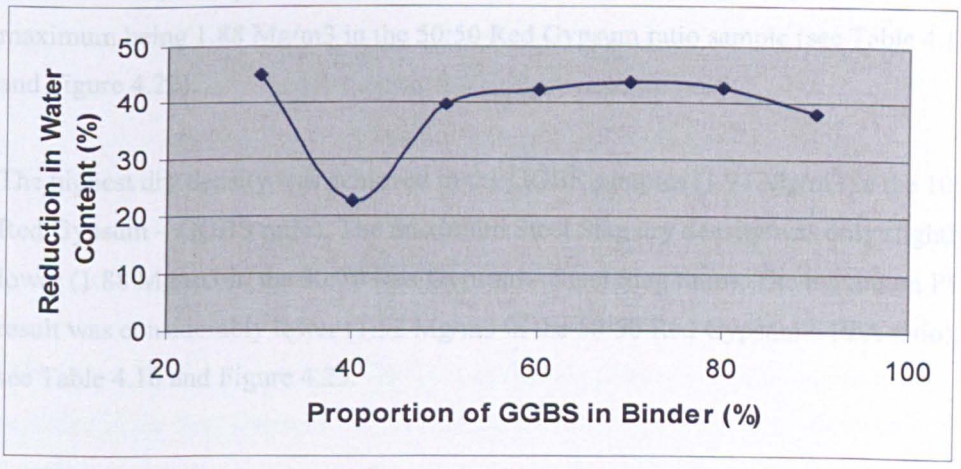



Figure 4.21, 2nd Phase Red Gypsum – GGBS water content reduction.

### 4.1.5 Combined density, strength and stiffness results

Table 4.4 shows a comparison of the peak results achieved in the different binder combinations, properties are discussed individually in the following sections.

Table 4.4, Combined binder density strength and stiffness results

| Material added to Red Gypsum for trials | Strength and Stiffness | | | | Density | | | |
|---|---|---|---|---|---|---|---|---|
| | RG: addition ratio | Water Content (%) | Max UCS (MPa) | Max Young's Modulus (MPa) | RG: addition ratio | Water Content (%) | Max Bulk Density (Mg/m3) | Max Dry Density (Mg/m3) |
| GGBS | 10-90 | 35.4 | 39.68 | - | 10-90 | 22.8 | 2.00 | 1.97 |
| PFA | 30-70 | 40.5 | 0.34 | 32.7 (@ 35.7% W.C) | 50-50 | 30.2 | 1.88 | 1.52 |
| Steel Slag | 30-70 | 18.56 | 4.51 | 681.1(@ 9.2% W.C) | 30-70 | 18.56 | 2.09 | 1.88 |

#### 4.1.5.1 Density

Figures 4.22 and 4.23 show a comparison of the bulk and dry densities achieved by the different binder mixes. Of the three investigations of materials added to Red Gypsum the highest bulk density achieved was in the Steel Slag mix (2.10 Mg/m3 in the 30:70 Red Gypsum – Steel Slag ratio). However, when GGBS was used, a relatively high bulk density was also achieved (2.00 Mg/m3 in the 10:90 Red Gypsum – GGBS ratio). Samples mixed with PFA achieved lower bulk densities, the maximum being 1.88 Mg/m3 in the 50:50 Red Gypsum ratio sample (see Table 4.10 and Figure 4.22).

The highest dry density was achieved in the GGBS samples (1.97 Mg/m3 in the 10:90 Red Gypsum – GGBS ratio). The maximum Steel Slag dry density was only slightly lower (1.88 Mg/m3 in the 30:70 Red Gypsum – Steel Slag ratio). The maximum PFA result was considerably lower (1.52 Mg/m3 in the 50:50 Red Gypsum – PFA ratio), see Table 4.10 and Figure 4.23.

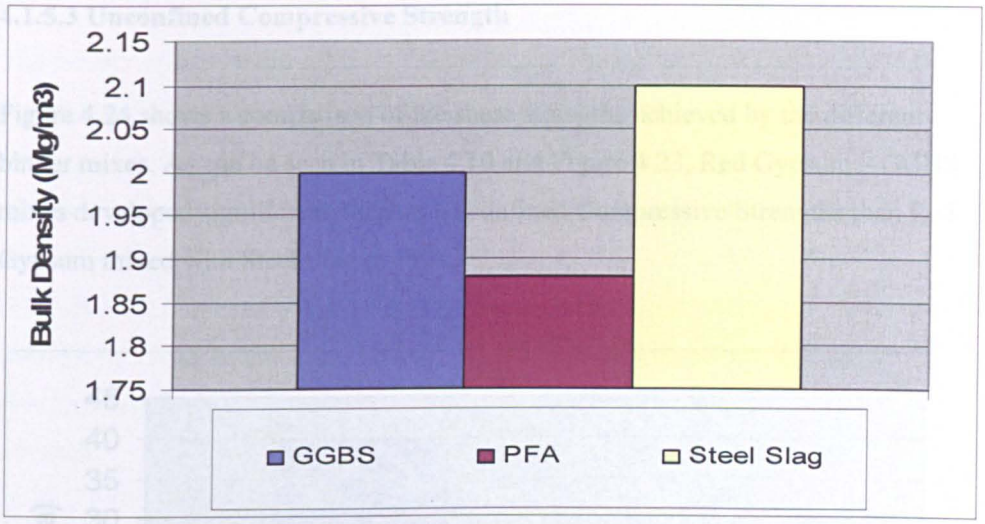



Figure 4.22, Combined binder bulk density results

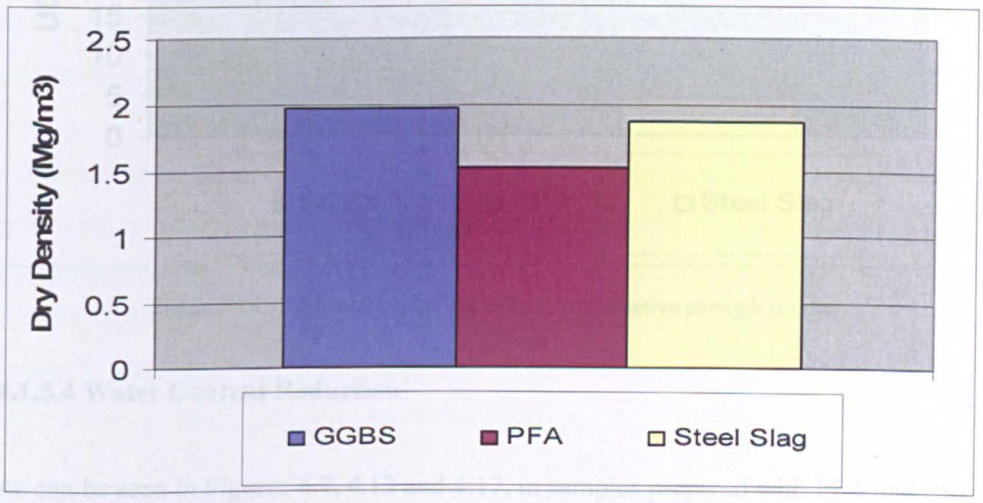



Figure 4.23, Combined binder dry density results.

### 4.1.5.2 Stiffness

Unfortunately no results are available for Red Gypsum – GGBS mixes. The maximum Young's Modulus recorded in the Red Gypsum PFA samples was 32.7 MPa in the 30:70 ratio sample. This is considerably lower than the maximum recorded in the Red Gypsum – Steel Slag mixes (681.1 MPa in the 10-90 ratio sample).

#### 4.1.5.3 Unconfined Compressive Strength

Figure 4.24 shows a comparison of the shear strengths achieved by the different binder mixes. As can be seen in Table 4.10 and Figure 4.23, Red Gypsum – GGBS mixes developed significantly higher Unconfined Compressive Strengths than Red Gypsum mixed with Steel Slag or PFA.

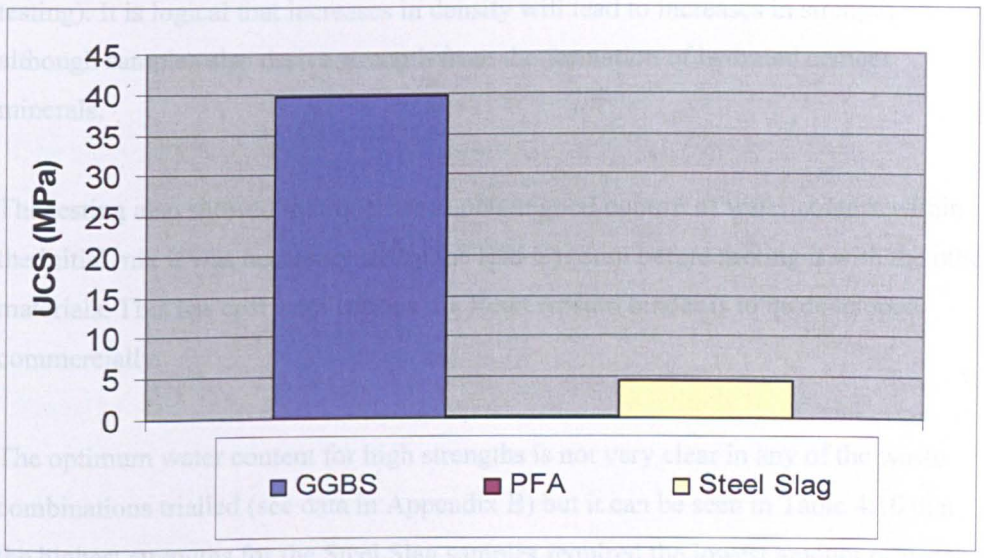



Figure 4.24, Combined binder unconfined compressive strength results.

#### 4.1.5.4 Water Content Reduction

As can be seen in Figures 4.7, 4.13 and 4.17, in samples prepared with PFA and Steel Slag those with higher initial water contents lost lower proportions of water than those with lower initial water contents. However, in the GGBS samples the opposite is the case.

#### 4.1.5.5 Conclusions of Physical Testing

In terms of the strengths and densities achieved in the various specimens it is clear that the binders made by mixing GGBS with Red Gypsum have the most desirable properties. The binders made with steel slag did show significant strengths but the PFA samples were very weak and would clearly be unsuitable for use as binders.

The density of the samples and the strengths achieved at 28 days are clearly linked, particularly in the cases of the steel slag and GGBS samples. In the case of the steel slag samples density and UCS both increased with increasing proportion of steel slag up to 70% after which values then dropped. In the case of the GGBS samples density and UCS generally increased with increasing proportions of GGBS, however, values could be seen to dip at 60% (this occurred in both phase 1 and 2 of the GGBS sample testing). It is logical that increases in density will lead to increases in strength although samples also derive strength from the formation of hydrated cement minerals.

The testing also showed that in order to obtain good control of water content within the initial mix it was necessary to dry the Red Gypsum before mixing it with the other materials. This has cost implications if a Red Gypsum binder is to be developed commercially.

The optimum water content for high strengths is not very clear in any of the waste combinations trialled (see data in Appendix B) but it can be seen in Table 4.10 that the highest strengths for the Steel Slag samples required the lowest amount of water (18.6%) followed by the GGBS samples (35.4%), the PFA samples required the most water (35.4%). The implications are that a greater amount of water would be required for mixing on site for the PFA and GGBS samples.

Despite the extra water required, from the results of the physical testing it is apparent that a combination of Red Gypsum and GGBS would make the best binder. In order to attain a greater control over water content using Red Gypsum in a dry powder form for mixing would be preferable if costs allow.

## 4.2. Mineralogy

### 4.2.1 Introduction

Mineralogical testing consisted of X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) analysis. Details of the testing techniques are contained in Chapter 3. Raw mineralogical testing data is contained in Appendix B. Table 4.5 and 4.6 show the samples upon which XRD and SEM tests were conducted.

Table 4.5, X –ray Diffraction Tests

| | | |
|---|---|---|
| Raw Materials | Red Gypsum | |
| | GGBS | |
| | Air Cooled Slag | |
| | PFA | |
| | Steel Slag | |
| Binder Mixes | Red Gypsum – PFA | 50:50 RG:PFA |
| | | 30:70 RG:PFA |
| | Red Gypsum – Steel Slag | 90:10 RG: Steel Slag |
| | | 70:30 RG : Steel Slag |
| | | 50:50 RG : Steel Slag |
| | | 30:70 RG : Steel Slag |
| | | 10:90 RG : Steel Slag |
| | GGBS | 70:30 RG : GGBS |
| | | 60:40 RG : GGBS |
| | | 50:50 RG : GGBS |
| | | 40:60 RG : GGBS |
| | | 30:70 RG : GGBS |
| | | 20:80 RG : GGBS |
| | | 10:90 RG : GGBS |

Table 4.6, SEM tests.

| Raw Materials | Binder Mixes |
|---|---|
| Red Gypsum | 80:20 RG : GGBS |
| GGBS | 30:60 RG : GGBS |

### 4.2.2 Raw Materials

In order to make proper analysis of cured binder mixes the raw materials used to make them were analysed first. In the case of X-ray analysis air cooled slag was tested in addition to GGBS. This was necessary as GGBS is glassy in nature and therefore X-ray amorphous.

#### 4.2.2.1 X-ray Diffraction Analysis

A complete set of XRD plots is contained in Appendix B, two plots are shown here for demonstration purposes (Figure 4.25).

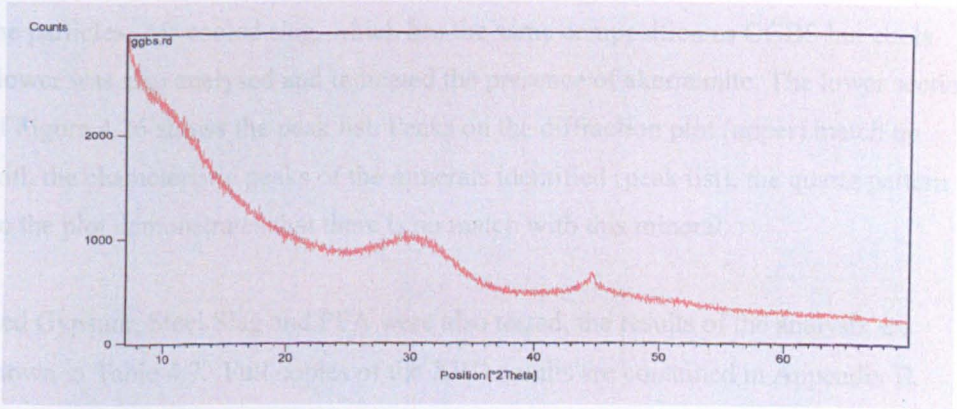



Figure 4.25, GGBS XRD plot.

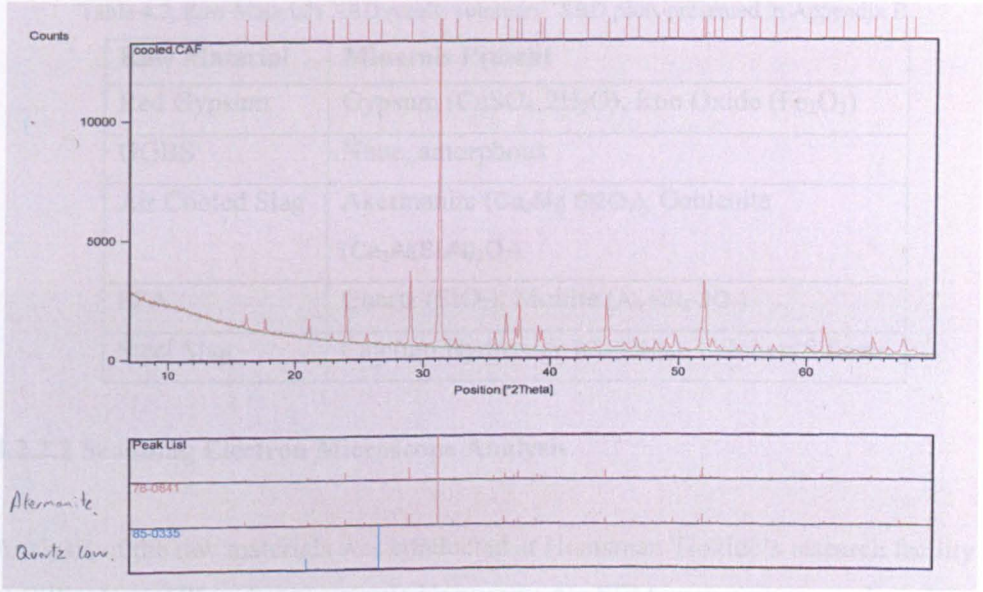



Figure 4.26, Air cooled slag XRD plot, lower section shows minerals identified.

Figures 4.25 and 4.26 show the X-ray diffraction patterns of GGBS and air cooled slag. As stated previously GGBS particles are glassy due to quick cooling in the production process and are therefore X-ray amorphous. This is shown clearly in Figure 4.25 as the refraction pattern gives no indication of the mineral composition of the particles. Air cooled slag, which has the same composition as GGBS but cools slower was also analysed and indicated the presence of akermanite. The lower section of Figure 4.26 shows the peak list. Peaks on the diffraction plot (upper) match up with the characteristic peaks of the minerals identified (peak list), the quartz pattern on the plot demonstrates that there is no match with this mineral.

Red Gypsum, Steel Slag and PFA were also tested, the results of the analysis are shown in Table 4.7. Full copies of the XRD results are contained in Appendix B.

Table 4.7, Raw Materials XRD results summary, XRD plots presented in Appendix B.

| Raw Material | Minerals Present |
|---|---|
| Red Gypsum | Gypsum ($CaSO_4.2H_2O$), Iron Oxide ($Fe_2O_3$) |
| GGBS | None, amorphous |
| Air Cooled Slag | Akermanite ($Ca_2Mg\ Si2O_7$), Gehlenite ($Ca_2Al(Si,Al)_2O_7$) |
| PFA | Quartz ($SiO_2$), Mullite ($Al_2+Si_2-2O_7$) |
| Steel Slag | Calcium Hydroxide ($Ca(OH)_2$), Calcium Silicate |

### 4.2.2.2 Scanning Electron Microscope Analysis

Analysis of the raw materials was conducted at Huntsman Tioxide's research facility at Billingham, UK and at Newcastle University. No SEM analysis was conducted on PFA and steel slag samples due to high costs. Where samples were tested at Newcastle University, point elemental analysis was conducted on the crystal grains. Readings from the point analysis were used to compare with later binder samples (see Appendix B for test data).

**Red Gypsum**

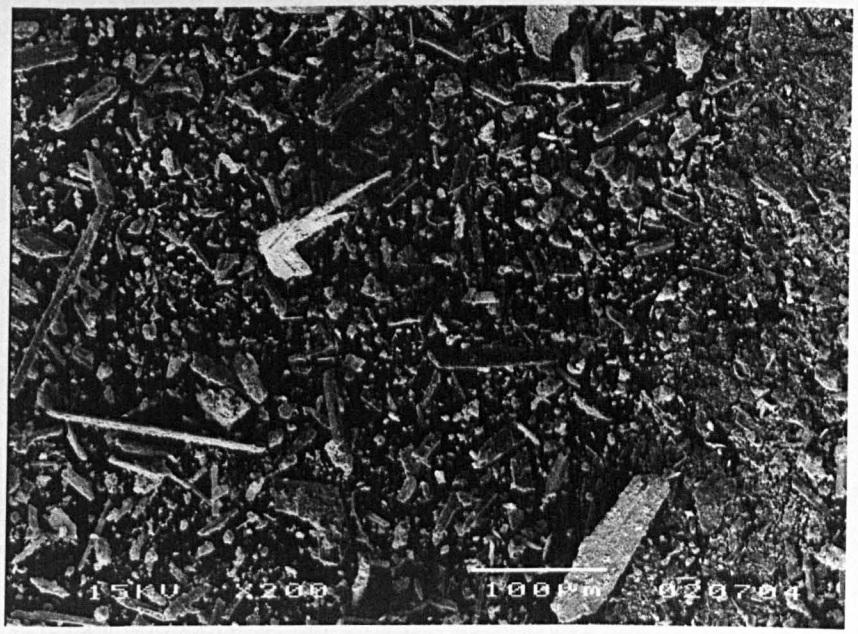



Figure 4.27, SEM image of Red Gypsum particles (Huntsman Tioxide).

Red Gypsum was analysed at Huntsman and at Newcastle University. Figure 4.27 shows that the Red Gypsum particles are needle like in shape and up to 200 microns long.

**GGBS**

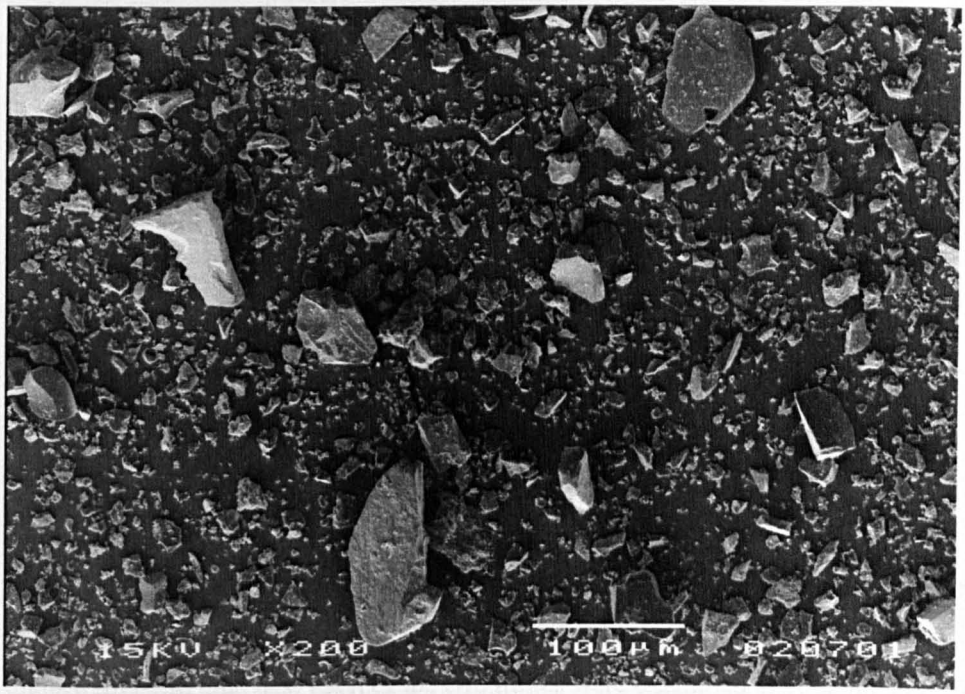



Figure 4.28, SEM image of GGBS particles.

Figure 4.28 shows that the GGBS particles were between 2 and 200 microns and angular to sub-rounded in shape.

### 4.2.3 Binder Mixes

#### 4.2.3.1 X-ray Diffraction Analysis

Copies of the binder mix XRD data are contained in Appendix B, a summary of the results are shown in Table 4.8.

Table 4.8, Summary of X-ray diffraction results (quartz $SiO_2$, thaumasite, ettringite $C_6AS_3H_{32}$, gypsum $CaSO_4 2H_2O$, rutile $TiO_2$)

| Binder | Ratio | Minerals identified |
|---|---|---|
| Red Gypsum – PFA | 50:50 RG:PFA | Quartz, thaumasite, ettringite, gypsum |
| | 30:70 RG:PFA | quartz, thaumasite, ettringite, gypsum |
| Red Gypsum – Steel Slag | 90:10 RG: Steel Slag | gypsum, thaumasite, ettringite |
| | 70:30 RG : Steel Slag | gypsum, thaumasite, ettringite |
| | 50:50 RG : Steel Slag | gypsum, thaumasite, ettringite |
| | 30:70 RG : Steel Slag | gypsum, thaumasite, ettringite |
| | 10:90 RG : Steel Slag | gypsum, thaumasite, ettringite, $TiO_2$ |
| Red Gypsum - GGBS | 70:30 RG : GGBS | gypsum, ettringite |
| | 60:40 RG : GGBS | gypsum, ettringite |
| | 50:50 RG : GGBS | gypsum, ettringite |
| | 40:60 RG : GGBS | gypsum, ettringite |
| | 30:70 RG : GGBS | gypsum, ettringite |
| | 20:80 RG : GGBS | gypsum, ettringite |
| | 10:90 RG : GGBS | gypsum, ettringite, rutile |

### 4.2.3.2 Scanning Electron Microscope Analysis

Figures 4.29 – 4.34 show SEM images of Red Gypsum – GGBS binder samples. Due to costs only two binder samples (80:20 RG : GGBS and 30:60 RG : GGBS) underwent SEM analysis. Each of the two mixes were examined at 100, 500 and 1500 times magnification, and point elemental analysis was conducted on the red spots shown in the Figures. In the 80:20 Red Gypsum : GGBS mix, at low magnification the it can be seen that the crystals are predominantly needle like gypsum, although some white rounded grains can also be seen. These are particles of GGBS, and some quartz is also present (see Figure 4.29). The same pattern is repeated at medium magnification (see Figure 4.30) where more of the matrix can be seen. Figure 4.31 shows the section at high magnification and much more of the amorphous matrix can be seen. Point elemental analysis was conducted on areas of the matrix and this indicated that the matrix was composed of assorted cement forming minerals including tridymite and di-calcium silicate.

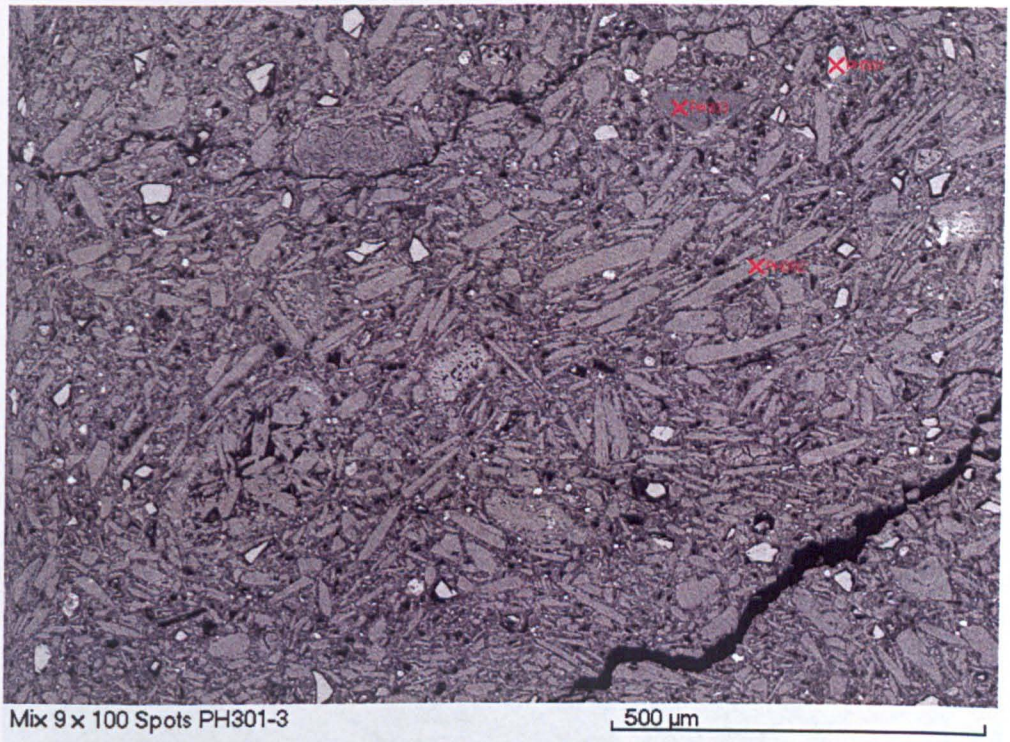



Figure 4.29, Polished section of 70:30 RG:GGBS mix low magnification, spots PH301 = GGBS, PH302 = gypsum, PH303 = quartz.

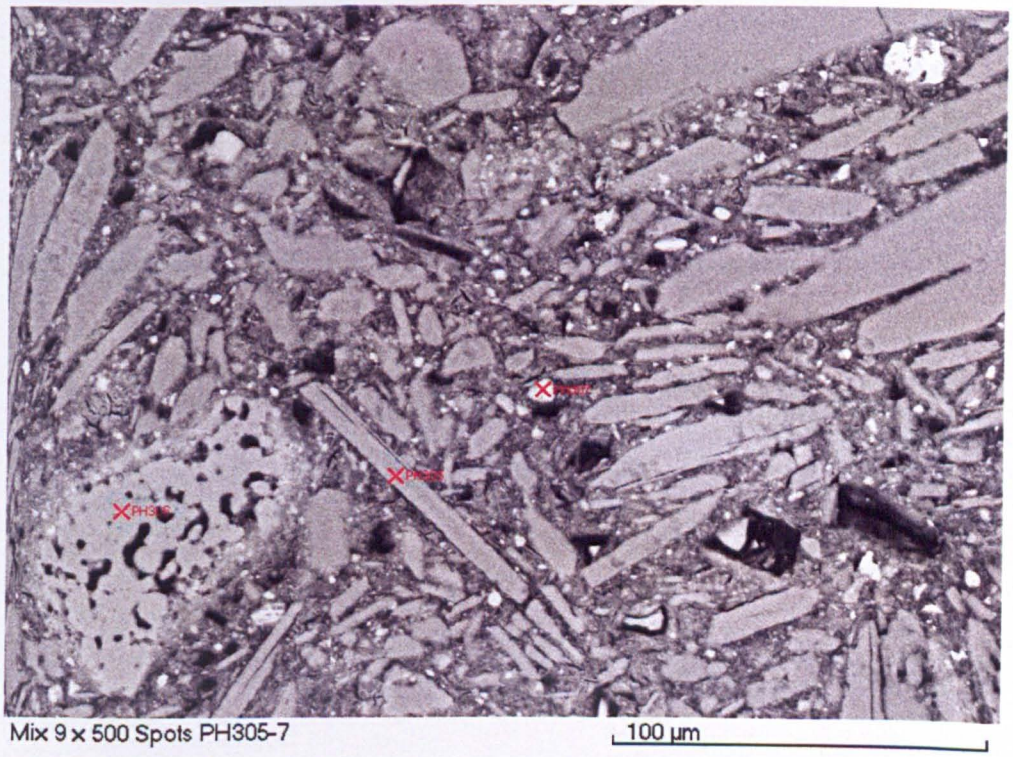



Figure 4.30, Polished section of 70:30 RG:GGBS mix medium magnification, spots PH305 = gypsum, PH306 = Lime, PH307 = GGBS.

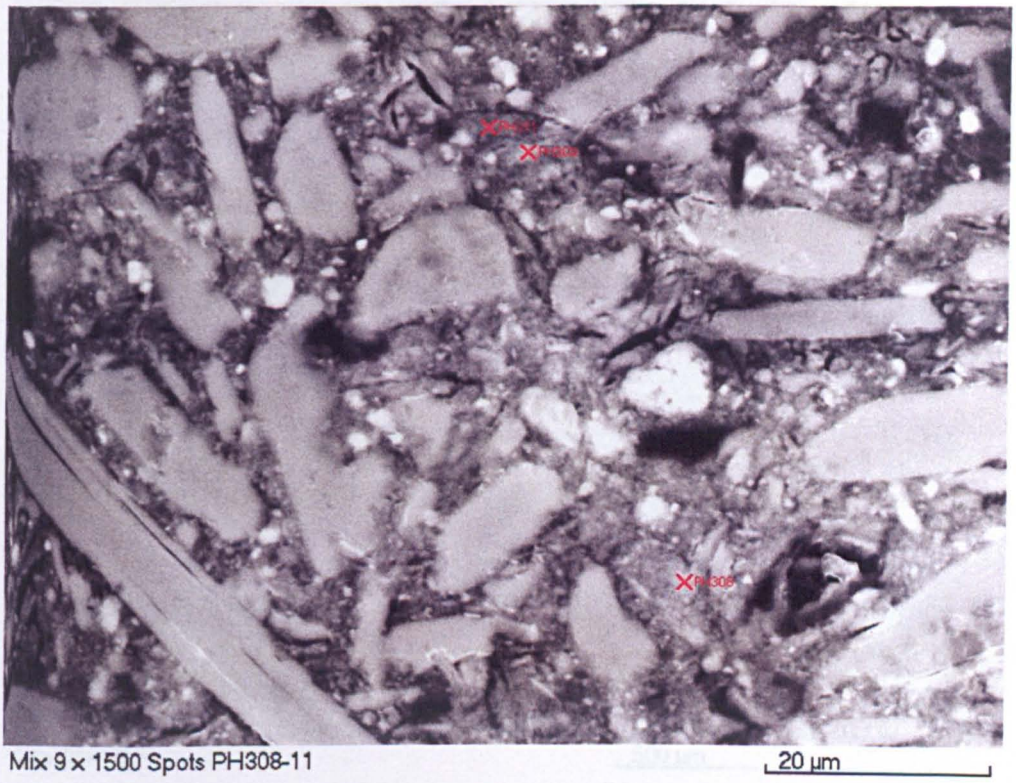



Figure 4.31, Polished section of 70:30 RG:GGBS mix high magnification

Figure 4.32 shows the 70:30 Red Gypsum : GGBS mix at low magnification. As anticipated more GGBS particles are clearly present in the section, although the gypsum crystals are still clearly identifiable. At medium magnification (Figure 4.33) there is clearly more amorphous matrix visible than the equivalent 80:20 image. At high magnification much more of the amorphous matrix can be seen.

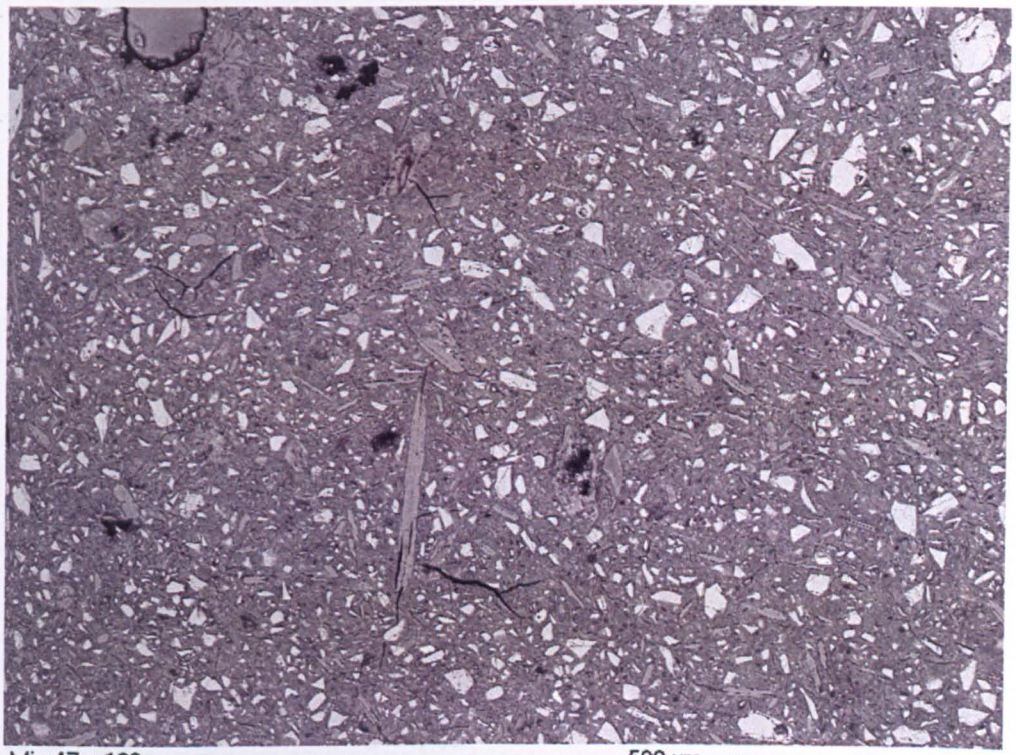



Figure 4.32, Polished section of 40:60 RG:GGBS mix low magnification, no point analysis conducted.

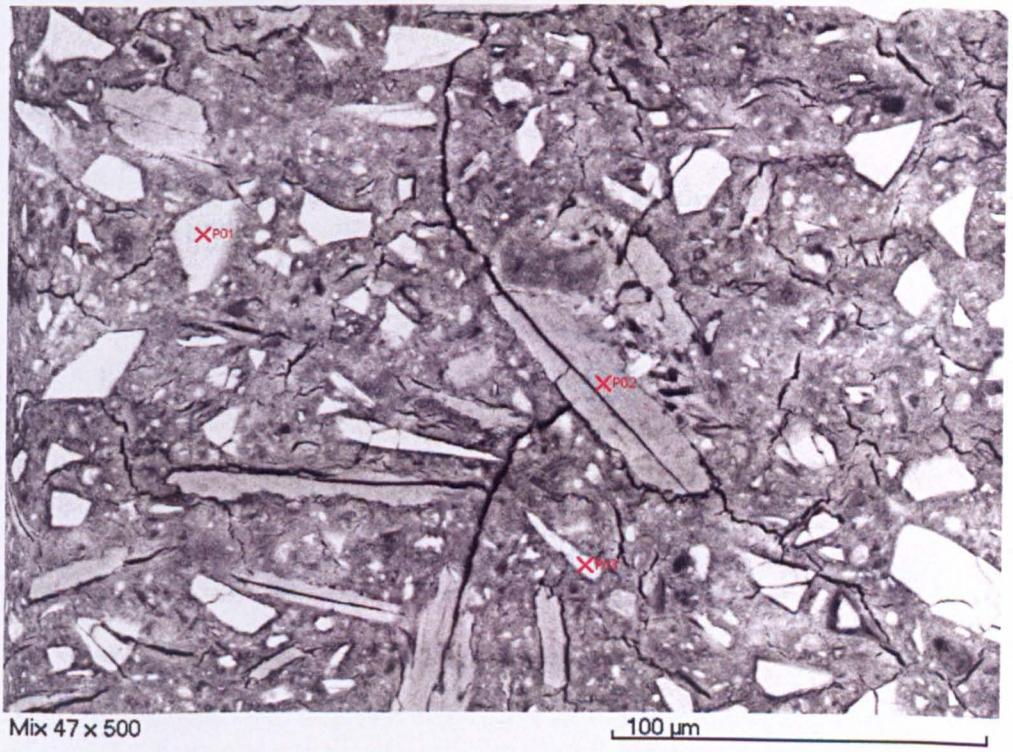



Figure 4.33, Polished section of 40:60 RG:GGBS mix medium magnification, spots P01 = GGBS, P02 = gypsum, P03 = GGBS .


Figure 4.34, Polished section of 40:60 RG:GGBS mix high magnification, spots P01&P02 = Di-Calcium Silicate, white areas are GGBS.

The results of the binder SEM testing are shown in Table 4.9.

Table 4.9, Summary of SEM analysis results on Red Gypsum : GGBS.

| Mix Ratio | Minerals Identified |
|---|---|
| 70:30 RG : GGBS | GGBS, gypsum, quartz, lime, and di-calcium silicate |
| 40:60 RG :GGBS | GGBS, gypsum and di-calcium silicate |

### 4.2.4 Mineral Results Comparison

Table 4.13 shows that despite different proportions of waste materials within the mixes tested, the results from the X-ray analysis of the binder combinations were almost identical. The Red Gypsum – PFA samples and the Red Gypsum – Steel Slag samples all contained quartz, thaumasite, ettringite and gypsum. The Red Gypsum – GGBS samples all indicated the presence of Gypsum and ettringite. Two samples were exceptions to this, 10:90 RG : Steel Slag and 10:90 RG : GGBS both exhibited the presence of Titanium Dioxide ($TiO_2$). This is attributed to residue from the $TiO_2$ production process contained in the Red Gypsum. Unfortunately the XRD analysis cannot quantify the amounts of ettringite present.

SEM analysis was only carried out on the Red Gypsum – GGBS samples. Table 4.9 summarises the results from the SEM tests. This demonstrates the presence of un-hydrated GGBS particles, gypsum, lime, quartz, and di-calcium silicate.

The presence of GGBS particles, gypsum, quartz and lime within the samples are all easy to explain as they are all components of the initial mix ingredients and it is understandable that some of these materials would be un-altered after the curing period of 28 days. The presence of these materials in the cured binder does not have any serious detrimental consequences on the strength of the binder as the strength results for these samples were high (see section 4.5).

All samples indicated the presence of ettringite. The presence of the ettringite within the samples is a possible cause for concern although during the testing programme no swelling of the samples was observed. Also it should be noted that whilst the X-ray diffraction analysis techniques used can identify the presence of ettringite it cannot quantify it and only small amounts may have formed, small quantities occur in all concretes. There is also the possibility that the formation of ettringite within the samples may in fact strengthen the material to an extent.

A greater concern is the indication of the presence of thaumasite within some of the samples. Thaumasite can greatly reduce the strength of cemented materials. This

material was only detected in the Steel Slag and PFA samples, both of which exhibited significantly lower strengths than the GGBS samples (see section 4.5)

Titanium dioxide ($TiO_2$) was detected in two of the samples, 10:90 RG : Steel Slag and 10:90 RG : GGBS. This material will almost certainly originate from within the Red Gypsum, and other than having a slight effect on the colour this material has not had a significant effect on the properties of the binder as can be seen from the strength results (Table 4.4).

### 4.3 Discussion and Conclusions

The Steel Slag samples exhibited the highest bulk densities and the GGBS samples the highest dry density (see Table 4.4). The dry density is more significant as it indicates the density of the solids within the samples. The PFA samples exhibited lower densities than the Steel Slag and GGBS samples. The density of the samples within the different combinations trialled indicates that higher strengths were developed at higher densities. Whilst in some applications lower densities are desirable (lightweight concrete for example) the strength of the binder is more important. In the case of lightweight concretes the type of aggregate has much more of an impact on the density achieved

The Red Gypsum – GGBS samples exhibited by far the greatest unconfined compressive strengths (see Table 4.4 and Figure 4.23) being nearly 10 times the strength achieved by the other combinations. It was also possible to measure Young's Modulus in the steel slag and PFA samples of these other binder combinations the steel slag samples exhibited much higher stiffness than the PFA samples (Table 4.4).

The X-ray diffraction mineralogical testing indicated that only the Red Gypsum – GGBS samples did not contain thaumasite. It was also possible to conduct SEM analysis on the Red Gypsum – GGBS samples, though this analysis did indicate the presence of hydrated cement minerals (di-calcium silicate) but unfortunately no comparison can be made with the other binders. The presence of thaumasite within the steel slag and PFA samples is one possible explanation for the significantly low strengths exhibited.

In the light of all of the results of the binder testing it was clear that the Red Gypsum – GGBS combination was by far the most effective binder, and whilst there was evidence to suggest that ettringite was present within the samples, this did not necessarily mean that samples made with the binder will swell.

The strongest ratio of the Red Gypsum – GGBS mix binders was 10:90 (39.68 MPa), although the 50:50 mix achieved over 70 % of this strength (28.63 MPa), (see Table 4.4). Given that one of the objectives of the research was to develop a binder to make use of large volumes of Red Gypsum, and that GGBS already has many applications within the construction industry, pragmatism suggests that using a 50:50 Red Gypsum – GGBS mix is the most appropriate combination to trial as a binder in engineering applications.

It would be desirable to conduct further testing on binders with PFA and Steel Slag additions possibly using other materials to boost the strength and stiffness. But for the purposes of this research it is felt that it would be more effective to only continue with Red Gypsum – GGBS.

# Chapter 5

# Soil Mixing Results

# 5. Soil Mixing Results

## 5.1 Physical Testing

The aims of the physical testing were to:

- Assess the potential strength and stiffness benefits of mixing a Red Gypsum based binder with a range of soils over a range of curing periods.
- Investigate the links between free water content, strain at failure, curing time, binder concentration, strength and stiffness.
- Observe the differences, if any, in the properties of samples cured in soaked and un-soaked conditions.
- Compare the performance of Red Gypsum binder with commercially available Portland cement.

The binder selected in chapter 4 (50:50 Red Gypsum – GGBS with 2% added lime) was mixed with 4 soils:

- Glacial Till
- Silty Sand (laboratory mixed)
- London Clay
- Irish Moss Peat

In the case of the Irish Moss Peat the testing programme was suspended at the preliminary stage after it became clear that significant strength gains would not be achieved. This is explained in the discussion section.

Details of the index and engineering properties of the soils can be seen in Chapter 3, along with the methods of test employed in this study.

### 5.1.1 Silty Sand

#### 5.1.1.1 Shear Strength

As can be seen in Figure 5.1 the addition of Red Gypsum – GGBS binder increased the strength of the silty sand progressively with curing time. Greater additions of binder caused greater increases in strength. For the Red Gypsum – GGBS samples the strength increased most rapidly between 0 and 56 days, after which the rate of strength increase reduced, though still increased until 112 days. The Portland cement control samples (20% concentration) increased in strength much more rapidly than the Red Gypsum – GGBS samples, with more than 50% of the strength increase occurring in the first 7 days. However, by 28 days the Portland cement and Red Gypsum shear strengths were approximately the same, though the plot indicates that the 20% Red Gypsum samples would achieve higher strengths than the Portland cement samples over longer curing periods.

The same pattern can be seen in the soaked curing samples as shown in Figure 5.2. However in the case of the soaked samples the 20% Portland cement control samples were stronger than the Red Gypsum – GGBS samples through to 112 days curing although the Red Gypsum – GGBS samples did achieve 80% of the strength of the Portland cement samples.

Table 5.1 shows that the dry cured samples achieved shear strengths approximately twice that of the soaked samples. It should be noted that there is significant variation in strengths between samples of the same concentration and curing time, and this is most likely to have been caused by minor variations in compaction and mixing.

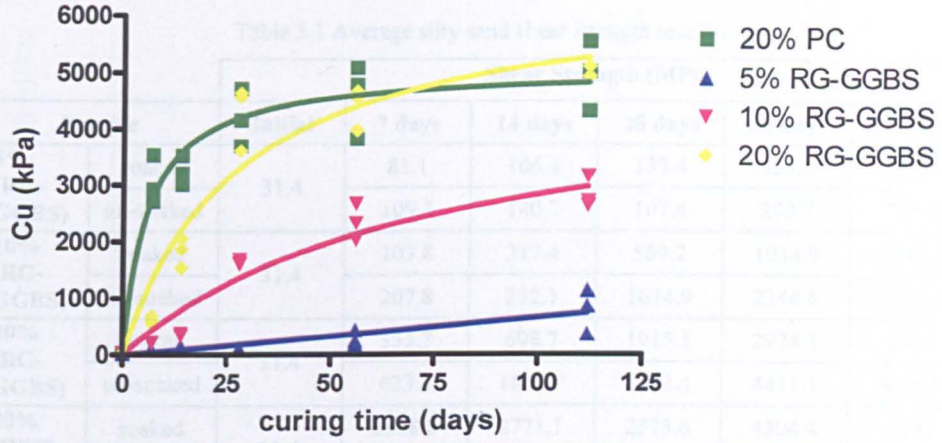



Figure 5.1, Silty sand shear strength results, un-soaked curing.

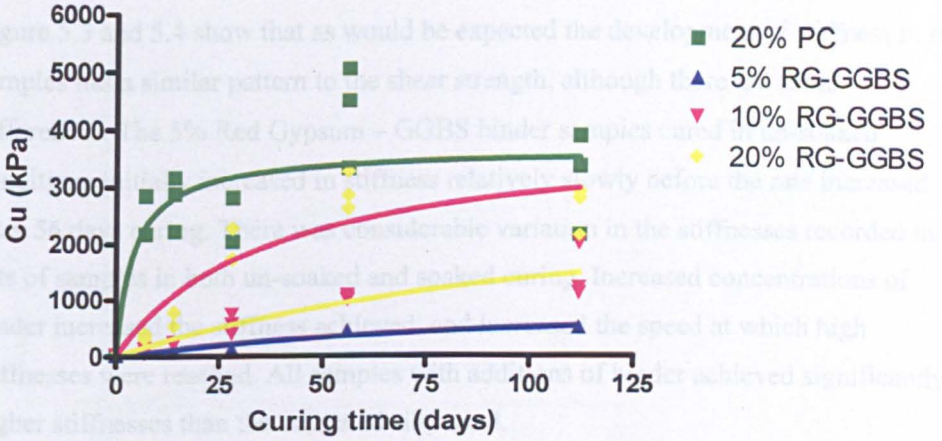



Figure 5.2, Silty sand shear strength results, soaked curing.

Table 5.1 Average silty sand shear strength results.

| Sample | | Shear Strength (MPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 31.4 | 81.1 | 106.4 | 133.4 | 457.2 | 515.2 |
| | un-soaked | | 109.3 | 140.7 | 107.4 | 292.7 | 796.5 |
| 10% (RG-GGBS) | soaked | 31.4 | 103.8 | 217.4 | 569.2 | 1034.9 | 1446.7 |
| | un-soaked | | 207.8 | 352.1 | 1634.9 | 2344.6 | 2840.9 |
| 20% (RG-GGBS) | soaked | 31.4 | 355.7 | 698.7 | 1915.1 | 2924.1 | 2621 |
| | un-soaked | | 623.3 | 1840.7 | 4282.4 | 4411.1 | 5000.7 |
| 20% (OPC) | soaked | 31.4 | 2528.8 | 2771.1 | 2573.6 | 4304.4 | 3142 |
| | un-soaked | | 3061.0 | 3257.1 | 4201.4 | 4577 | 4974.9 |

### 5.1.1.2 Young's Modulus

Figure 5.3 and 5.4 show that as would be expected the development of stiffness in the samples has a similar pattern to the shear strength, although there are some differences. The 5% Red Gypsum – GGBS binder samples cured in un-soaked conditions initially increased in stiffness relatively slowly before the rate increased after 56 days curing. There was considerable variation in the stiffnesses recorded in sets of samples in both un-soaked and soaked curing. Increased concentrations of binder increased the stiffness achieved, and increased the speed at which high stiffnesses were reached. All samples with additions of binder achieved significantly higher stiffnesses than the untreated silty sand.

As with the shear strength results the Portland cement samples achieved high stiffnesses within 7 days. However, the Red Gypsum – GGBS samples achieved 80% of the Portland cement sample stiffness by 56 days and approximately the same stiffness by 112 days.

Samples cured in un-soaked conditions achieved up to 120% of the stiffness of the samples cured in soaked conditions.

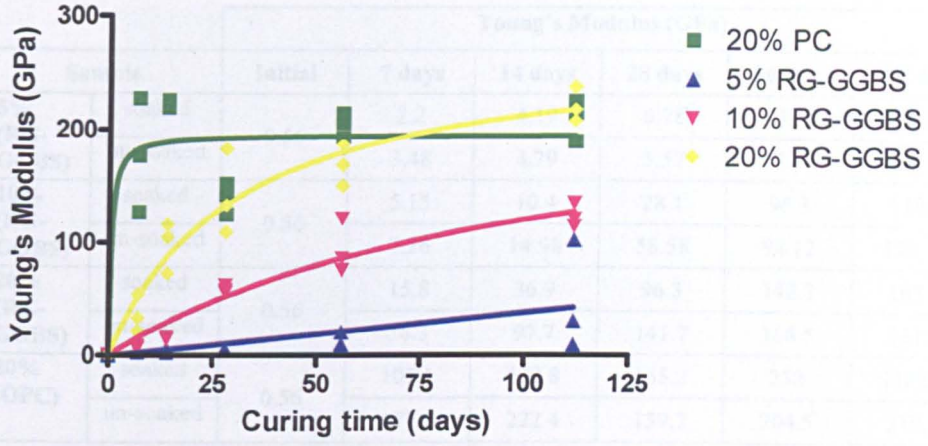



Figure 5.3, Silty sand, Stiffness results, un-soaked curing.

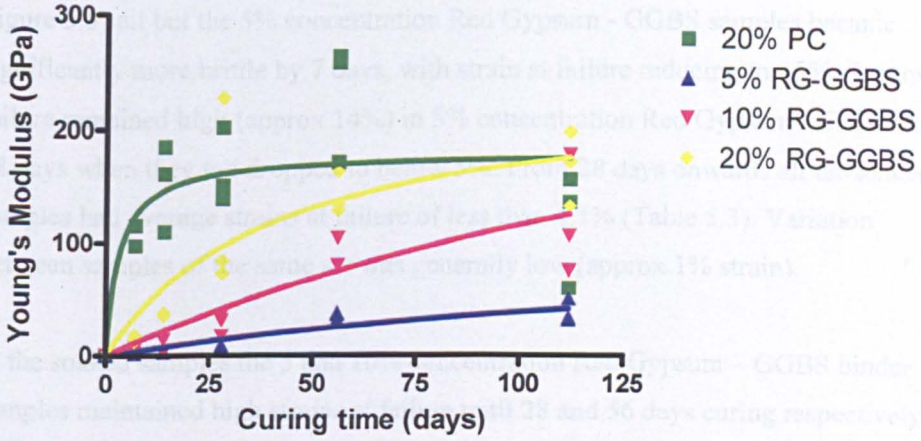



Figure 5.4, Silty sand, stiffness results, soaked curing.

Table 5.2, Average silty sand stiffness results.

| Sample | | Young's Modulus (GPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 0.56 | 2.2 | 4.19 | 6.78 | 35.76 | 38.85 |
| | un-soaked | | 3.48 | 4.79 | 5.57 | 13.63 | 47.7 |
| 10% (RG-GGBS) | soaked | 0.56 | 5.15 | 10.4 | 28.1 | 96.3 | 119.8 |
| | un-soaked | | 7.26 | 14.98 | 58.58 | 94.12 | 123.23 |
| 20% (RG-GGBS) | soaked | 0.56 | 15.8 | 36.9 | 96.3 | 142.7 | 167.7 |
| | un-soaked | | 36.3 | 97.7 | 141.7 | 168.5 | 221.7 |
| 20% (OPC) | soaked | 0.56 | 107.1 | 152.8 | 165.2 | 232 | 118.04 |
| | un-soaked | | 177.6 | 222.4 | 139.7 | 204.5 | 210.1 |

### 5.1.1.3 Strain at Failure

Untreated silty sand samples failed at between 15 and 20% strain, behaving in a plastic manner. When samples were cured in un-soaked conditions (as can be seen in Figure 5.5) all but the 5% concentration Red Gypsum - GGBS samples became significantly more brittle by 7 days, with strain at failure reducing to <5%. Strains to failure remained high (approx 14%) in 5% concentration Red Gypsum – GGBS until 28 days when they too dropped to below 5%. From 28 days onwards all un-soaked samples had average strains at failure of less than 4.1% (Table 5.3). Variation between samples of the same set was generally low (approx 1% strain).

In the soaked samples the 5 and 10% concentration Red Gypsum – GGBS binder samples maintained high strains at failure until 28 and 56 days curing respectively. Also there was variation in excess of 10% within samples of the same set.

In both soaked and un-soaked curing conditions the 20% Red Gypsum – GGBS samples exhibited very similar strains at failure to the Portland cement samples, being only slightly smaller (approx 1%).

Samples cured in soaked conditions exhibited lower strains at failure in the long term (from 56 days onwards).

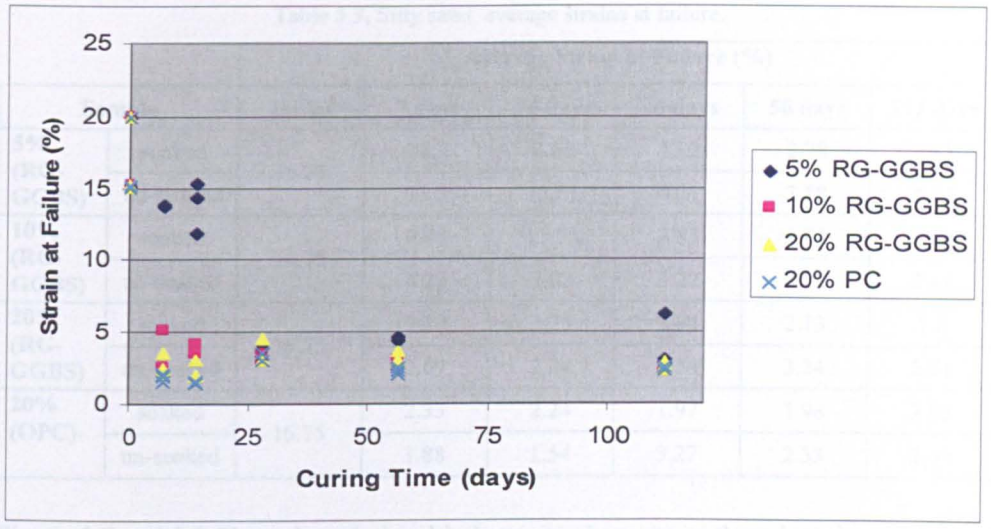



Figure 5.5, Un-soaked Silty sand Strain at failure.

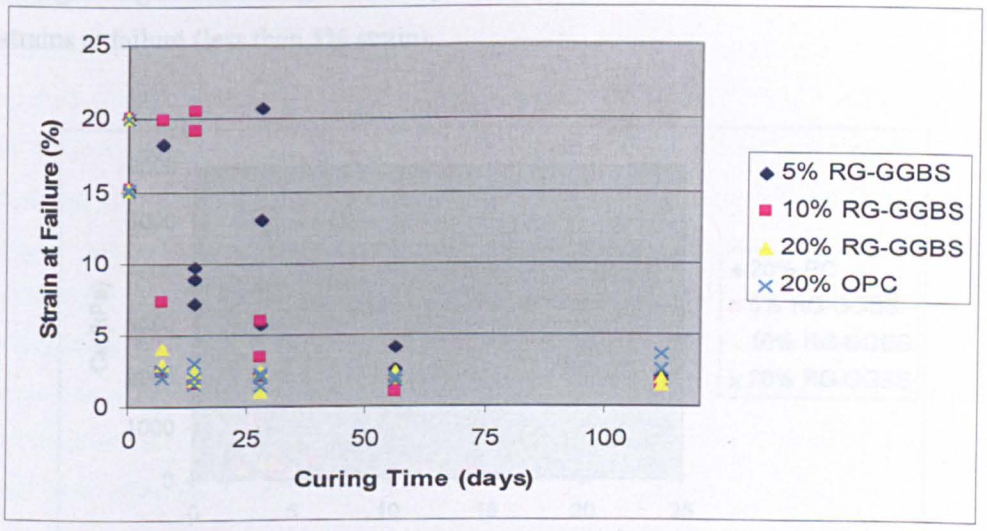



Figure 5.6, Soaked Silty sand strain at failure.

Table 5.3, Silty sand, average strains at failure.

| Sample | | Average Strain at Failure (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 16.75 | 18.3 | 8.65 | 13.2 | 2.78 | 1.83 |
| | un-soaked | | 13.3 | 13.81 | 4.11 | 3.88 | 4.05 |
| 10% (RG-GGBS) | soaked | 16.75 | 9.89 | 13.73 | 3.83 | 1.33 | 1.5 |
| | un-soaked | | 4.29 | 3.92 | 3.22 | 2.75 | 2.44 |
| 20% (RG-GGBS) | soaked | 16.75 | 3.29 | 2.25 | 1.94 | 2.13 | 1.8 |
| | un-soaked | | 2.69 | 2.34 | 3.54 | 3.24 | 2.61 |
| 20% (OPC) | soaked | 16.75 | 2.33 | 2.24 | 1.97 | 1.98 | 2.93 |
| | un-soaked | | 1.88 | 1.54 | 3.27 | 2.33 | 2.49 |

Figure 5.7 and 5.8 Show the relationship between shear strength and strain at failure. The plots show that in both soaked and un-soaked curing conditions, as would be expected, there is a strong relationship between the strain at failure and shear strength. High shear strengths are only exhibited by samples that also exhibit low strains at failure (less than 5% strain).

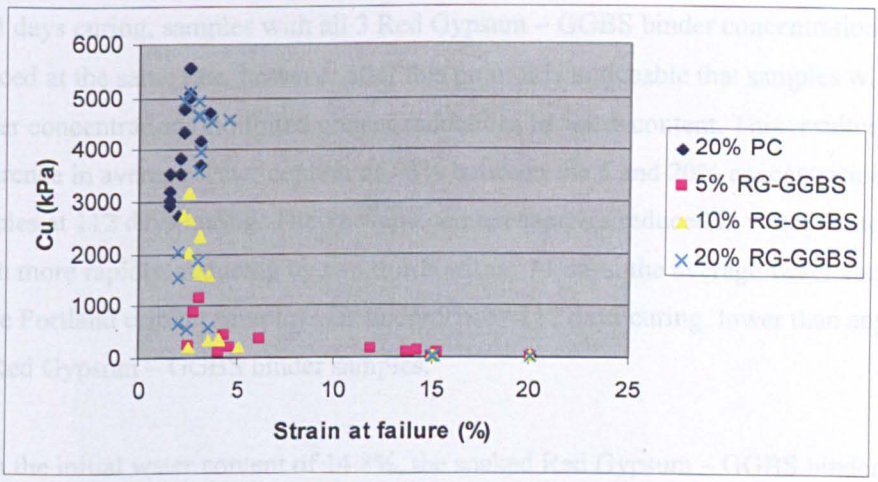



Figure 5.7 Shear strength vs strain at failure of un-soaked silty sand samples.

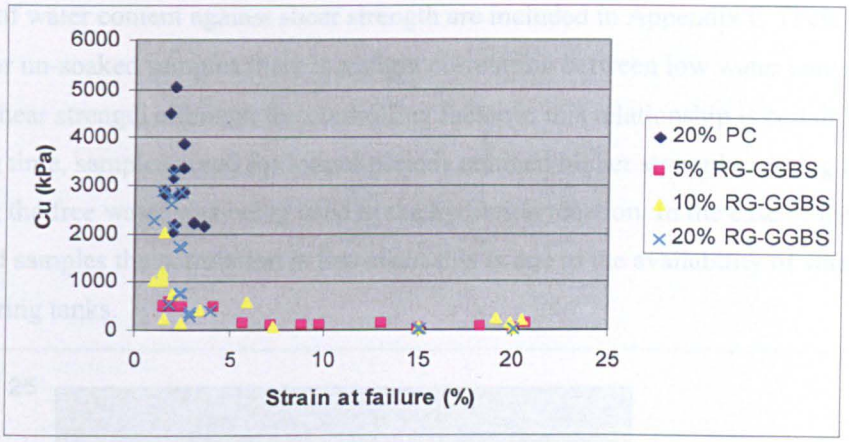



Figure 5.8 Shear strength vs strain at failure of soaked silty sand samples.

### 5.1.1.4 Water Content

Water content was measured post strength testing to make an assessment of the amount of water used during hydration. All samples started with an initial water content of 14.8%. In the case of the Red Gypsum – GGBS samples cured in un-soaked conditions this then began to drop steadily (see Figure 5.9 and Table 5.4). Up to 14 days curing, samples with all 3 Red Gypsum – GGBS binder concentrations reduced at the same rate, however after this point it is noticeable that samples with higher concentrations exhibited greater reductions in water content. This resulted in a difference in average water content of >3% between the 5 and 20% concentration samples at 112 days curing. The Portland cement samples reduced in water content much more rapidly, reducing by two thirds within 14 days, the average water content of the Portland cement samples was under 9% by 112 days curing, lower than any of the Red Gypsum – GGBS binder samples.

From the initial water content of 14.8%, the soaked Red Gypsum – GGBS binder samples immediately increased in water content (see Figure 5.10 and Table 5.4) to 15.8, 19.1 and 19% for 5, 10 and 20% concentrations respectively at 7 days although it can be seen in Figure 6.8 there was some variability in the water contents of the different sample sets. At 112 days the water contents for the 5, 10 and 20% samples were 16.6, 17.3 and 18.4% respectively. By comparison the Portland cement samples cured in soaked conditions maintained a relatively stable water content, varying by only 1.5% over the course of 112 days.

Plots of water content against shear strength are included in Appendix C These show that for un-soaked samples there is a slight correlation between low water content and high shear strength although the controlling factor in this relationship is certainly curing time, samples cured for longer periods attained higher strengths, during the curing the free water was being used in the hydration reaction. In the case of the soaked samples the correlation is less clear, this is due to the availability of water in the curing tanks.

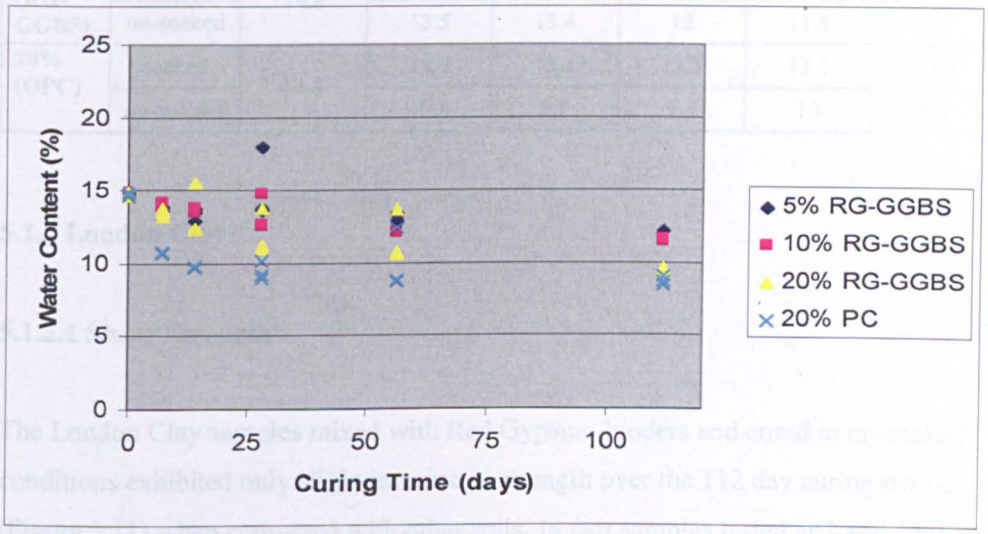



Figure 5.9 Un-soaked silty sand water contents

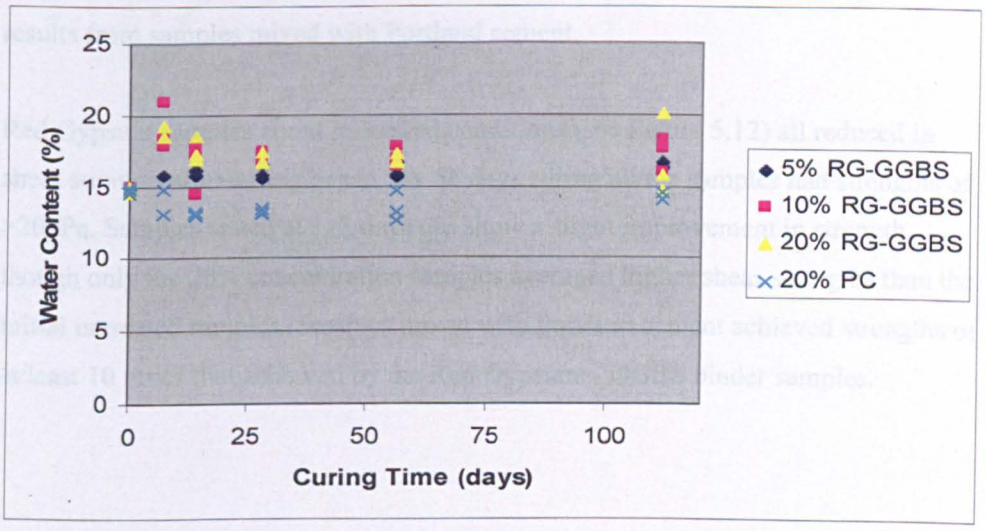



Figure 5.10 soaked silty sand water contents.

Table 5.4, Silty SAND average water contents.

| Sample | | Average Water Contents (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 14.8 | 15.8 | 16 | 16 | 16 | 16.6 |
| | un-soaked | | 13.8 | 13.4 | 13.8 | 13.2 | 13.2 |
| 10% (RG-GGBS) | soaked | 14.8 | 19.1 | 16.5 | 16.9 | 17 | 17.3 |
| | un-soaked | | 14 | 13.6 | 13.3 | 12.1 | 11.5 |
| 20% (RG-GGBS) | soaked | 14.8 | 19 | 17.6 | 16.9 | 16.9 | 18.1 |
| | un-soaked | | 13.5 | 13.4 | 12 | 11.6 | 9.5 |
| 20% (OPC) | soaked | 14.8 | 13.9 | 13.1 | 13.3 | 13.5 | 14.3 |
| | un-soaked | | 10.8 | 9.7 | 9.5 | 10 | 8.8 |

### 5.1.2 London Clay

#### 5.1.2.1 Shear Strength

The London Clay samples mixed with Red Gypsum binders and cured in un-soaked conditions exhibited only slight increase in strength over the 112 day curing period (Figure 5.11) when compared with other soils. In fact samples tested at 7 and 14 days reduced in shear strength by up to 40kPa. Samples tested at 56 days did show an increase in shear strength though this increase is not significant when compared to results from samples mixed with Portland cement.

Red Gypsum samples cured in soaked conditions (see Figure 5.12) all reduced in shear strength after curing began. By 56 days curing all the samples had strengths of >20kPa. Samples tested at 112 days did show a slight improvement in strength, though only the 20% concentration samples averaged higher shear strengths than the initial untreated samples. Samples mixed with Portland cement achieved strengths of at least 10 times that achieved by the Red Gypsum – GGBS binder samples.

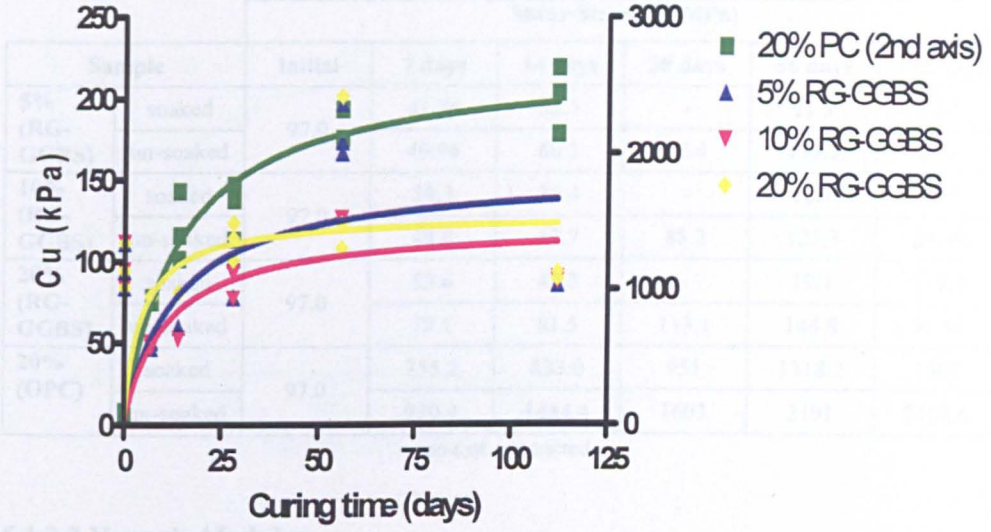



Figure 5.11, Un-soaked London clay, Shear strengths.

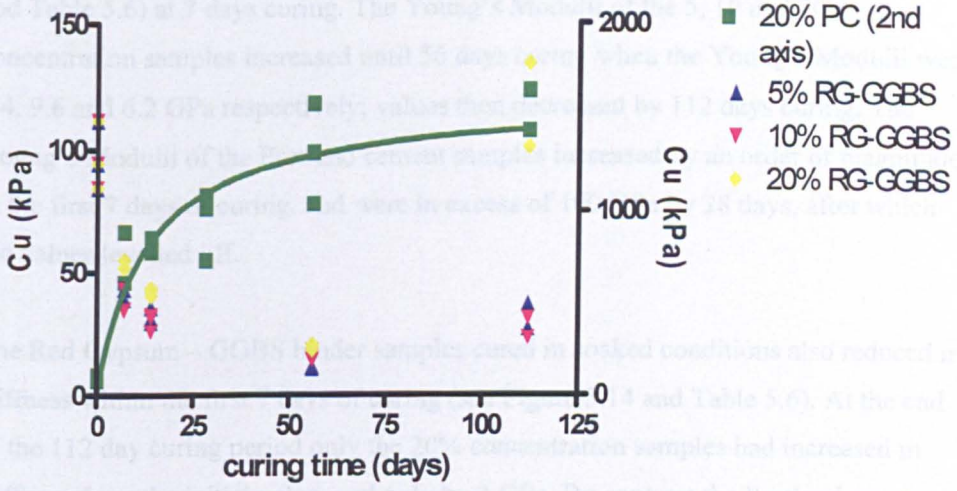



Figure 5.12, Soaked London Clay shear strengths.

Table 5.5, London Clay, average shear strengths.

| Sample | | Shear Strength (MPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 97.0 | 41.36 | 32.5 | - | 11.3 | 32.1 |
| | un-soaked | | 49.96 | 60.3 | 93.4 | 179.7 | 84.7 |
| 10% (RG-GGBS) | soaked | 97.0 | 39.2 | 30.4 | - | 16.1 | 27.5 |
| | un-soaked | | 48.8 | 52.7 | 88.2 | 125.3 | 89.39 |
| 20% (RG-GGBS) | soaked | 97.0 | 53.6 | 42.3 | - | 19.4 | 117.4 |
| | un-soaked | | 79.1 | 81.5 | 113.1 | 144.8 | 91.84 |
| 20% (OPC) | soaked | 97.0 | 755.2 | 823.0 | 951 | 1318.2 | 1507 |
| | un-soaked | | 930.9 | 1454.4 | 1603 | 2191 | 2408.6 |

- no test conducted

### 5.1.2.2 Young's Modulus

Starting at initial Young's Modulus of 4.29 GPa upon mixing, the Red Gypsum - GGBS un-soaked samples reduced in stiffness by approximately half (see Figure 5.13 and Table 5.6) at 7 days curing. The Young's Modulii of the 5, 10 and 20% concentration samples increased until 56 days curing when the Young's Modulii were 7.4, 9.6 and 6.2 GPa respectively; values then decreased by 112 days curing. The Young's Modulii of the Portland cement samples increased by an order of magnitude in the first 7 days of curing, and were in excess of 100 GPa by 28 days, after which the values levelled off.

The Red Gypsum – GGBS binder samples cured in soaked conditions also reduced in stiffness within the first 7 days of curing (see Figure 5.14 and Table 5.6). At the end of the 112 day curing period only the 20% concentration samples had increased in stiffness from the initial value, and only by 3 GPa. By contrast the Portland cement samples increased to 42 GPa after 7 days, and were in excess of 70 GPa after 112 days curing.

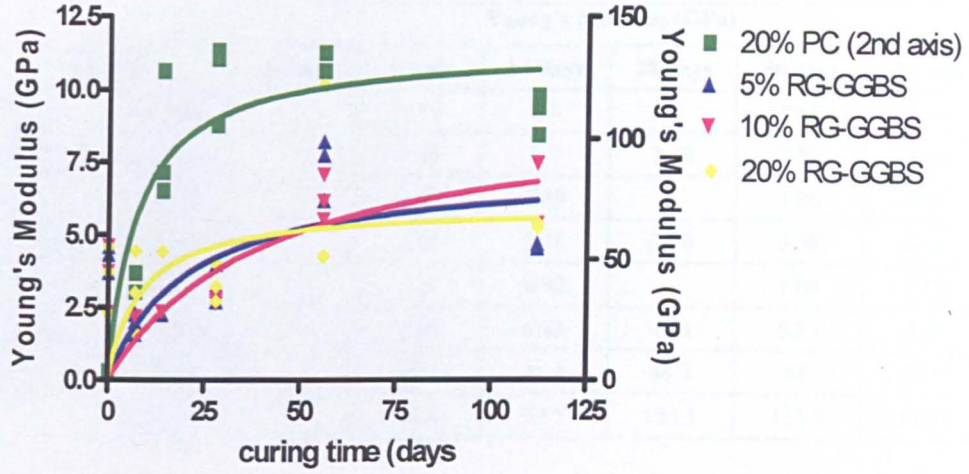



Figure 5.13, Un-soaked London Clay, Young's Modulus.

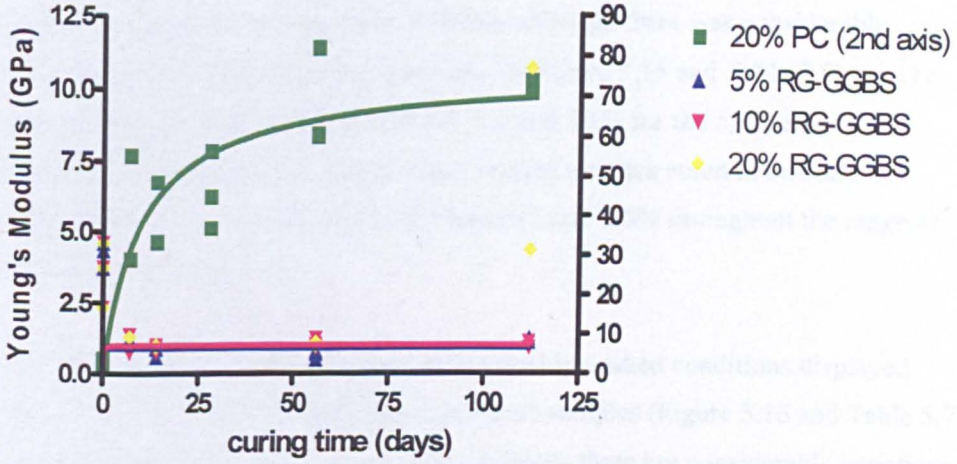



Figure 5.14, Soaked London Clay, Young's Modulus.

Table 5.6, London Clay, average Young's Modulus.

| Sample | | Young's Modulus (GPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 4.29 | 1.19 | 0.8 | - | 0.67 | 1.16 |
| | un-soaked | | 1.96 | 2.3 | 3.32 | 7.39 | 4.63 |
| 10% (RG-GGBS) | soaked | 4.29 | 1.05 | 0.89 | - | 1.06 | 0.99 |
| | un-soaked | | 1.97 | 2.36 | 2.79 | 9.58 | 6.42 |
| 20% (RG-GGBS) | soaked | 4.29 | 1.3 | 0.98 | - | 1.04 | 7.52 |
| | un-soaked | | 3.05 | 4.43 | 4.03 | 6.23 | 5.34 |
| 20% (OPC) | soaked | 4.29 | 42.3 | 41.2 | 46.3 | 68 | 72.5 |
| | un-soaked | | 40.8 | 97.7 | 125.1 | 133.5 | 110.4 |

### 5.1.2.3 Strain at Failure

The strain at failure for the initial untreated sample was 7.4%. Samples mixed with Red Gypsum – GGBS binder and cured in un-soaked conditions did not show significant change in average strain at failure although there was considerable variation between samples of the same set (see Figure 5.15 and Table 5.7). At 112 days the average strain at failure was 7.0, 6.2 and 5.1% for the 5, 10 and 20% concentrations respectively. The Portland cement samples cured in un-soaked conditions failed at average strains of between 2 and 2.5% throughout the range of curing periods (Table 5.7).

The Red Gypsum – GGBS binder samples cured in soaked conditions displayed strains at failure higher than the initial untreated samples (Figure 5.16 and Table 5.7). As with the samples cured in un-soaked conditions there are considerable variations between samples of the same set. Portland cement samples cured in soaked conditions failed at average strains of between 1.3 and 3% throughout the range of curing periods (Table 5.7).

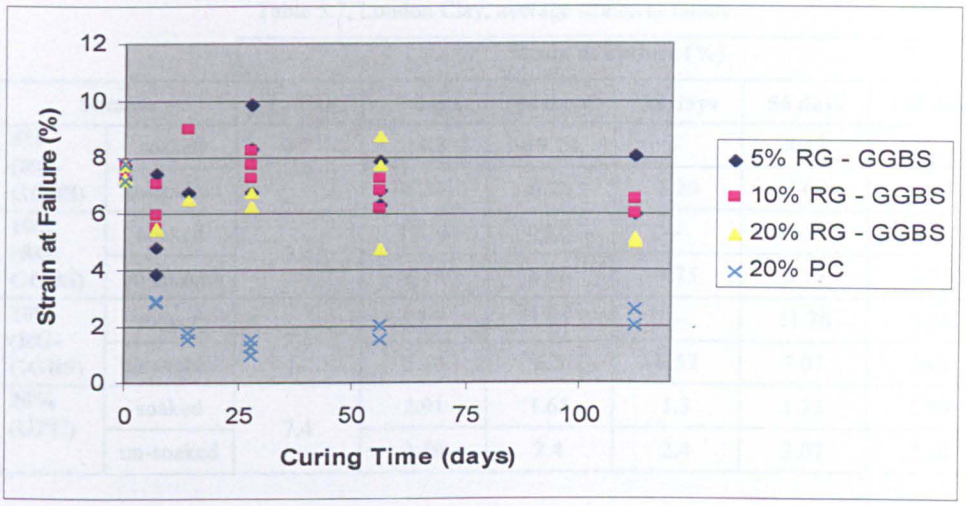



Figure 5.15, Un-soaked London Clay, Strain at Failure.

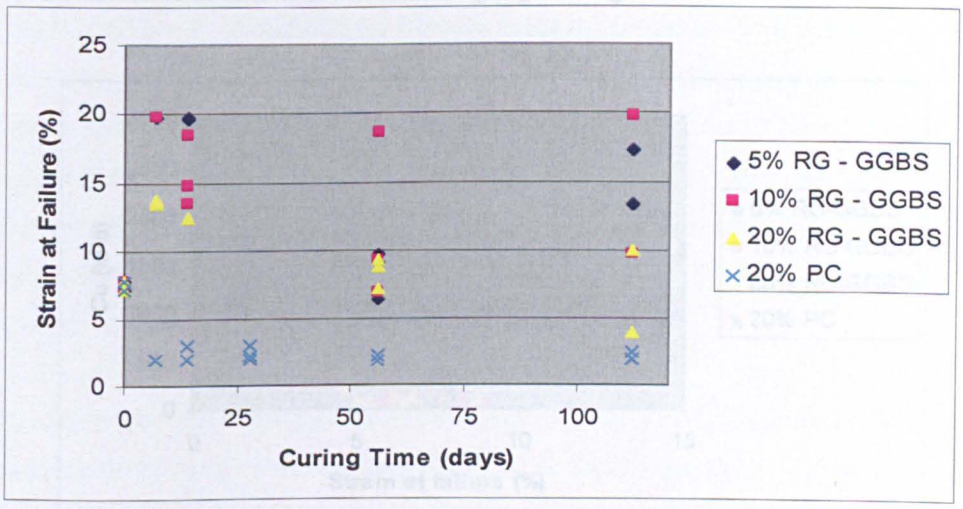



Figure 5.16, Soaked London Clay, Strain at Failure.

Table 5.7, London Clay, average strains at failure.

| Sample | | Strain at Failure (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 7.4 | 19.8 | 19.74 | - | 8.43 | 15.4 |
| | un-soaked | | 5.37 | 6.76 | 8.29 | 7.61 | 7.04 |
| 10% (RG-GGBS) | soaked | 7.4 | 19.78 | 15.6 | - | 11.64 | 14.61 |
| | un-soaked | | 6.16 | 8.98 | 7.75 | 6.76 | 6.25 |
| 20% (RG-GGBS) | soaked | 7.4 | 13.7 | 12.5 | - | 11.76 | 6.98 |
| | un-soaked | | 5.48 | 6.5 | 6.57 | 7.07 | 5.07 |
| 20% (OPC) | soaked | 7.4 | 2.91 | 1.65 | 1.3 | 1.73 | 2.63 |
| | un-soaked | | 2.49 | 2.4 | 2.4 | 2.07 | 2.30 |

Figures 5.17 and 5.18 show the relationship between shear strength and strain at failure of London Clay samples. As with the Silty sand samples there is a strong relationship between shear strength and strain at failure with only sample exhibiting strains at failure of less than 5% achieving high strengths.

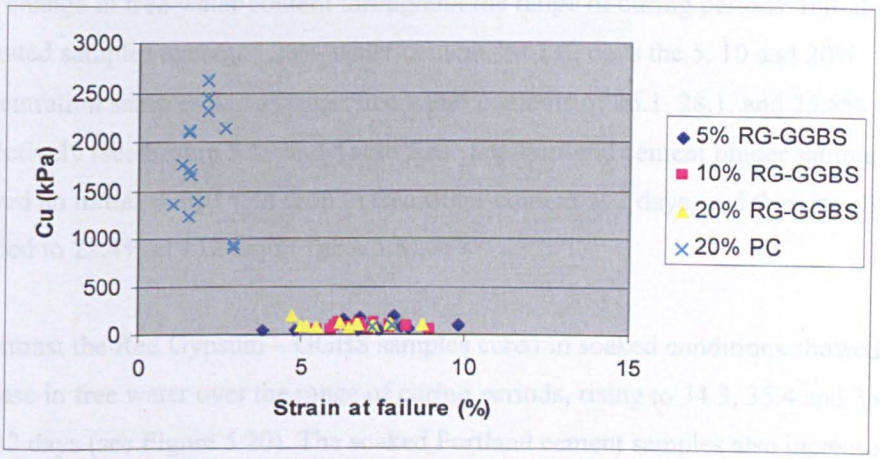



Figure 5.17 Shear strength vs strain at failure of un-soaked London Clay samples.

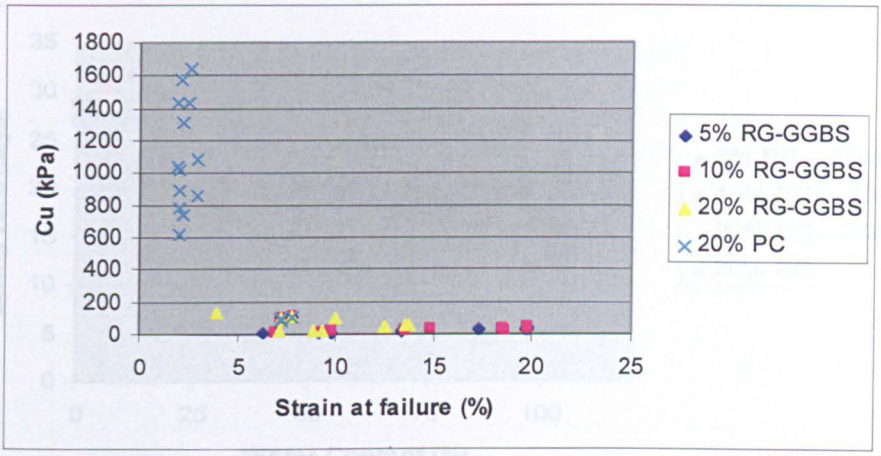



Figure 5.18 Shear strength vs strain at failure of soaked London Clay samples.

### 5.1.2.4 Water Content

The Red Gypsum – GGBS binder samples cured in un-soaked conditions showed little change in free water content throughout the range of curing periods. Initial untreated samples averaged 28% water content. At 112 days the 5, 10 and 20% concentration samples had average free water contents of 26.1, 28.1, and 25.8% respectively (see Figure 5.19 and Table 5.8). The Portland cement binder samples showed an initial sharp (4%) drop in free water content at 7 days, and then steadily reduced to 21.4% at 112 days (Table 5.8).

In contrast the Red Gypsum – GGBS samples cured in soaked conditions showed an increase in free water over the range of curing periods, rising to 34.3, 35.4 and 33.6% by 112 days (see Figure 5.20). The soaked Portland cement samples also increased in water content during the range of curing periods although not as much as the Red Gypsum – GGBS binder samples; rising to 32.6% by 122 days curing (Table 5.8).

Plots of water content against shear strength are included in Appendix C. These show that in both soaked and un-soaked samples there is a strong correlation between low water contents and higher strengths.

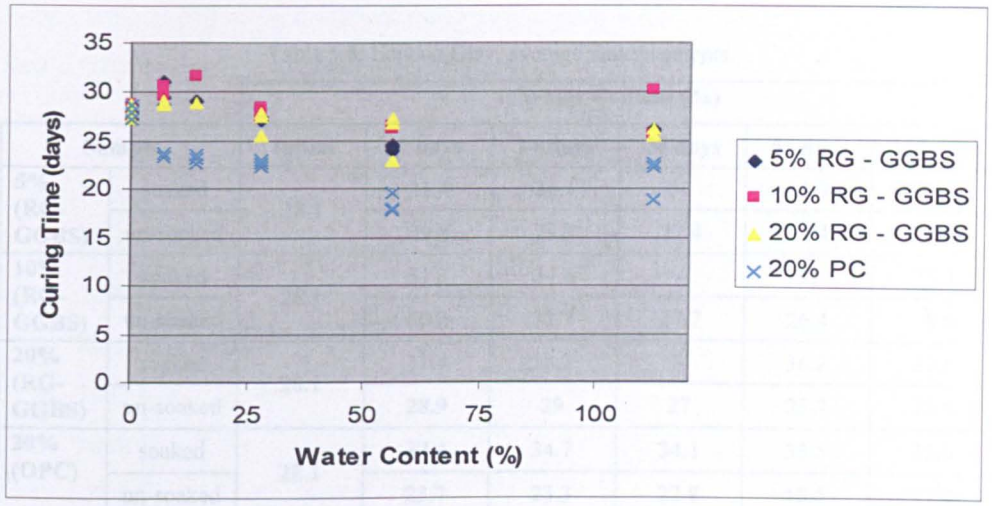



Figure 5.19, un-soaked London Clay, water contents.

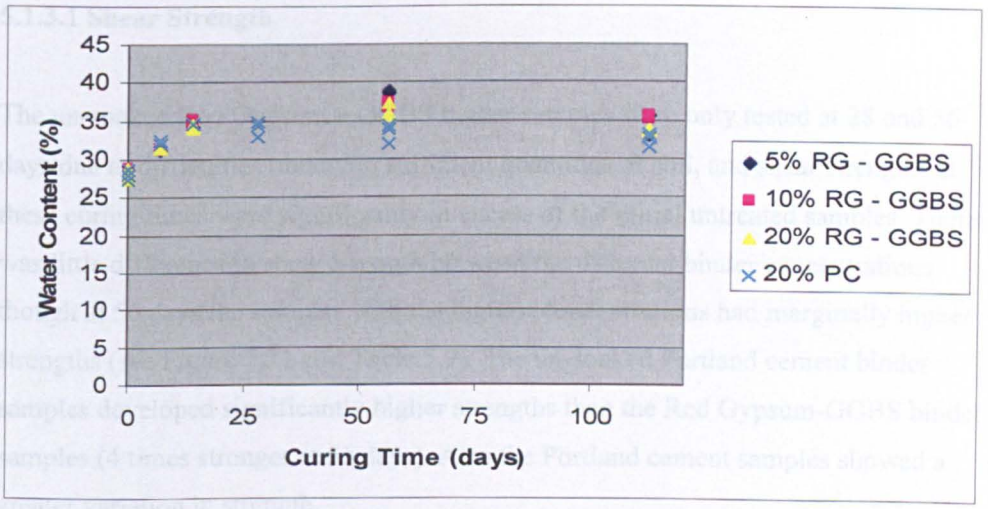



Figure 5.20, soaked London Clay, water contents.

Table 5.8, London Clay, average water contents.

| Sample | | Water Content (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 28.1 | 31.4 | 34.1 | - | 38.7 | 34.3 |
| | un-soaked | | 29.8 | 29.2 | 27.4 | 24.4 | 26.1 |
| 10% (RG-GGBS) | soaked | 28.1 | 31.3 | 34.6 | - | 37 | 35.4 |
| | un-soaked | | 30.2 | 31.7 | 27.7 | 26.4 | 28.0 |
| 20% (RG-GGBS) | soaked | 28.1 | 31.4 | 34.2 | - | 36.2 | 33.6 |
| | un-soaked | | 28.9 | 29 | 27 | 25.7 | 25.8 |
| 20% (OPC) | soaked | 28.1 | 32.4 | 34.7 | 34.1 | 33.5 | 32.6 |
| | un-soaked | | 23.7 | 23.3 | 22.8 | 18.5 | 21.4 |

### 5.1.3 Glacial Till

#### 5.1.3.1 Shear Strength

The un-soaked Red Gypsum – GGBS binder samples were only tested at 28 and 56 days due to difficulties obtaining sufficient quantities of soil, and shear strengths at these curing times were significantly in excess of the initial untreated samples. There was little difference in shear strength between the different binder concentrations though at 56 days the samples with the highest concentrations had marginally higher strengths (see Figure 5.21 and Table 5.9). The un-soaked Portland cement binder samples developed significantly higher strengths than the Red Gypsum-GGBS binder samples (4 times stronger at 56 days). Also the Portland cement samples showed a greater variation in strength.

The soaked Red Gypsum – GGBS binder samples were tested at the full range of curing times, the results are shown in Figure 5.22. All concentrations showed a rise in average shear strengths after 7 days compared to the initial untreated samples. However, strengths did not increase significantly until 28 days. Samples with higher concentrations of binder achieved higher shear strengths, and at 112 days the average shear strengths were 160, 379 and 891 kPa for the 5, 10 and 20% concentrations

respectively. The Portland cement samples achieved higher average strengths than the Red Gypsum – GGBS samples (see Table 5.9)

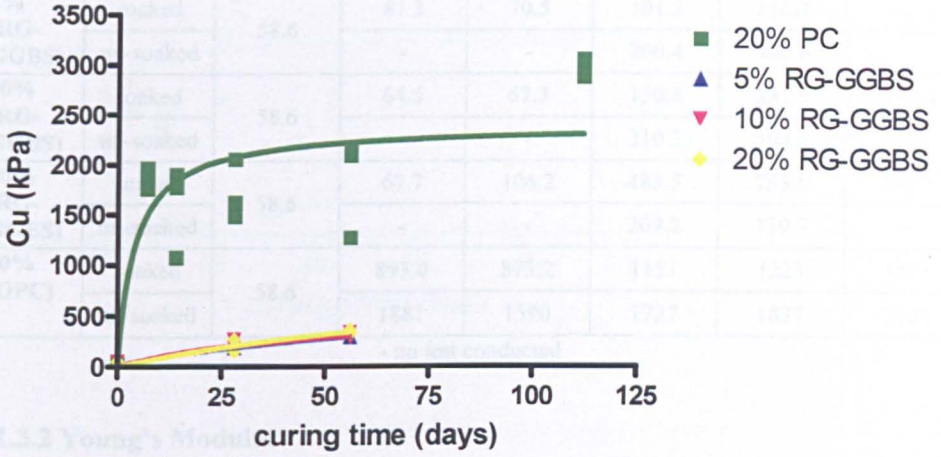



Figure 5.21, Un-soaked Glacial Till, Shear Strengths.

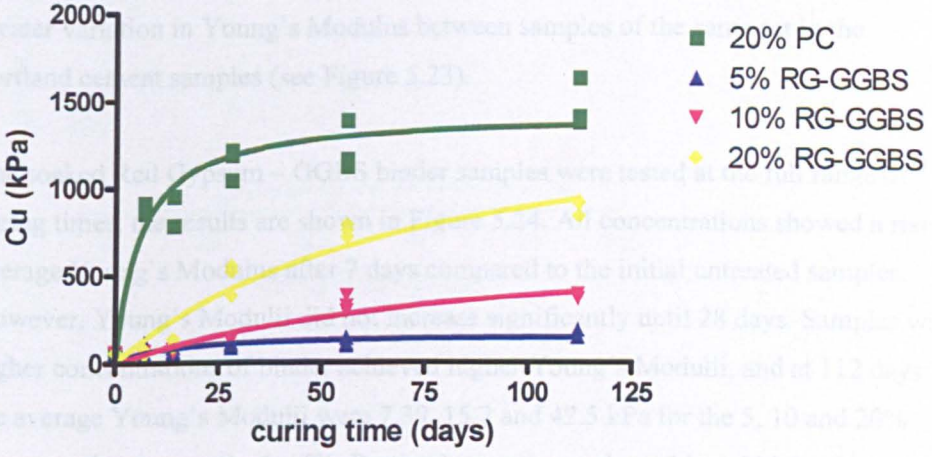



Figure 5.22, Soaked Glacial Till, Shear Strengths.

Table 5.9, Glacial Till, average shear strengths.

| Sample | | Shear Strength (MPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 58.6 | 81.3 | 70.5 | 101.5 | 116.3 | 160.7 |
| | un-soaked | | - | - | 200.4 | 300.6 | - |
| 10% (RG-GGBS) | soaked | 58.6 | 64.5 | 67.3 | 130.8 | 331.9 | 379.4 |
| | un-soaked | | - | - | 210.3 | 304.4 | - |
| 20% (RG-GGBS) | soaked | 58.6 | 67.7 | 106.2 | 485.5 | 753.6 | 891.5 |
| | un-soaked | | - | - | 209.2 | 339.7 | - |
| 20% (OPC) | soaked | 58.6 | 893.0 | 873.2 | 1151 | 1223 | 1475 |
| | un-soaked | | 1881 | 1590 | 1727 | 1837 | 2967 |

- no test conducted

### 5.1.3.2 Young's Modulus

The un-soaked Red Gypsum – GGBS binder samples increased in average Young's Modulus from the initial value of 1.03 GPa to 21.1, 16.6, and 21.8 GPa for the 5, 10 and 20% concentrations respectively at 56 days curing. This compares to 106.5 GPa for the un-soaked Portland cement binder samples at 56 days. There was considerably greater variation in Young's Modulus between samples of the same set in the Portland cement samples (see Figure 5.23).

The soaked Red Gypsum – GGBS binder samples were tested at the full range of curing times, the results are shown in Figure 5.24. All concentrations showed a rise in average Young's Modulus after 7 days compared to the initial untreated samples. However, Young's Modulii did not increase significantly until 28 days. Samples with higher concentrations of binder achieved higher Young's Modulii, and at 112 days the average Young's Modulii were 7.39, 15.2 and 42.5 kPa for the 5, 10 and 20% concentrations respectively. The Portland cement samples achieved higher Young's Modulii (see Table 5.10).

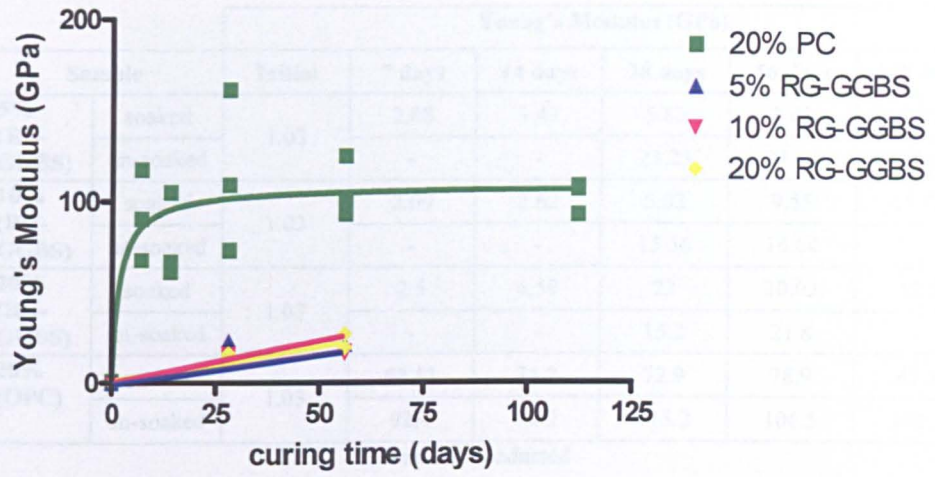



Figure 5.23, Un-soaked Glacial Till, Young's Modulus.

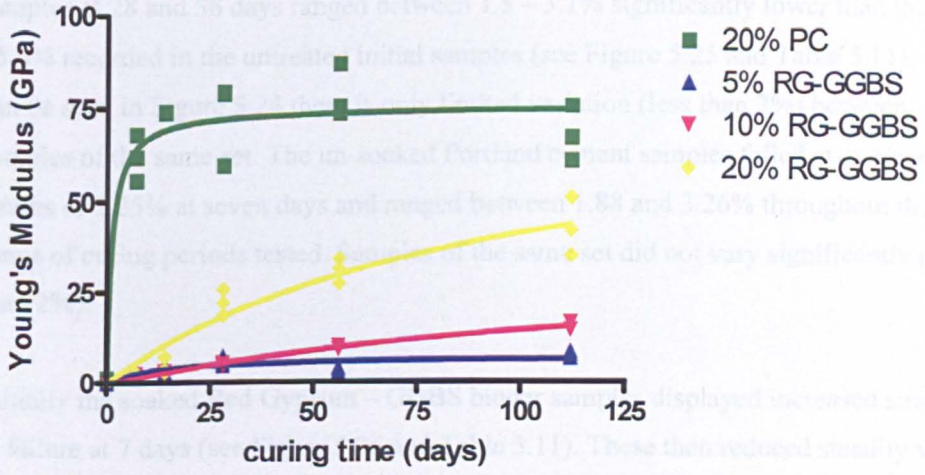



Figure 5.24 Soaked Glacial Till, Young's Modulus.

Table 5.10, Glacial Till average Young's Modulii.

| Sample | | Young's Modulus (GPa) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 1.03 | 2.88 | 3.43 | 5.82 | 3.63 | 7.39 |
| | un-soaked | | - | - | 23.23 | 21.14 | - |
| 10% (RG-GGBS) | soaked | 1.03 | 2.09 | 2.62 | 5.02 | 9.55 | 15.23 |
| | un-soaked | | - | - | 15.56 | 16.64 | - |
| 20% (RG-GGBS) | soaked | 1.03 | 2.5 | 4.59 | 22 | 30.02 | 42.5 |
| | un-soaked | | - | - | 15.2 | 21.8 | - |
| 20% (OPC) | soaked | 1.03 | 62.13 | 74.2 | 72.9 | 78.9 | 67.8 |
| | un-soaked | | 92.4 | 78.2 | 115.2 | 106.5 | 103.2 |

- no test conducted

### 5.1.3.3 Strain at Failure

The average strains at failure recorded in the un-soaked Red Gypsum – GGBS binder samples at 28 and 56 days ranged between 1.5 – 3.1% significantly lower than the 15.6% recorded in the untreated initial samples (see Figure 5.25 and Table 5.11). As can be seen in Figure 5.24 there is only limited variation (less than 2%) between samples of the same set. The un-soaked Portland cement samples failed at average strains of 2.25% at seven days and ranged between 1.88 and 3.26% throughout the range of curing periods tested. Samples of the same set did not vary significantly (less than 2%).

Initially the soaked Red Gypsum – GGBS binder samples displayed increased strains at failure at 7 days (see Figure 5.26 and Table 5.11). These then reduced steadily with increasing curing time, at 112 days the average strains at failure for the 5, 10 and 20% concentration samples being 6.9, 4.6, and 2.89% respectively. Samples with lower concentrations of binder displayed higher strains at failure. In contrast the soaked Portland cement samples became brittle much more quickly, displaying average strains at failure of 2% by 7 days. The values then ranged between 1.7 and 2.4 throughout the range of curing periods tested.

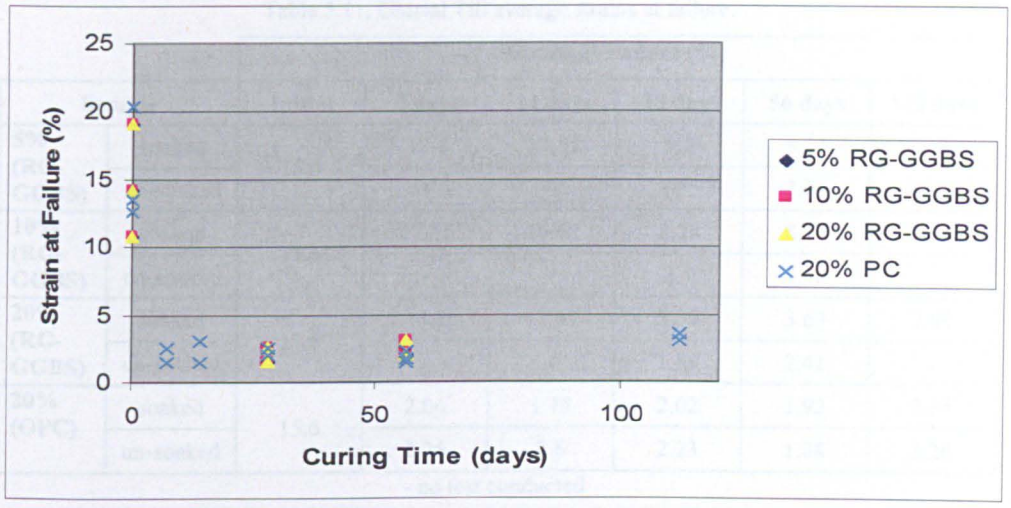



Figure 5.25, Un-soaked Glacial Till, strain at failure.

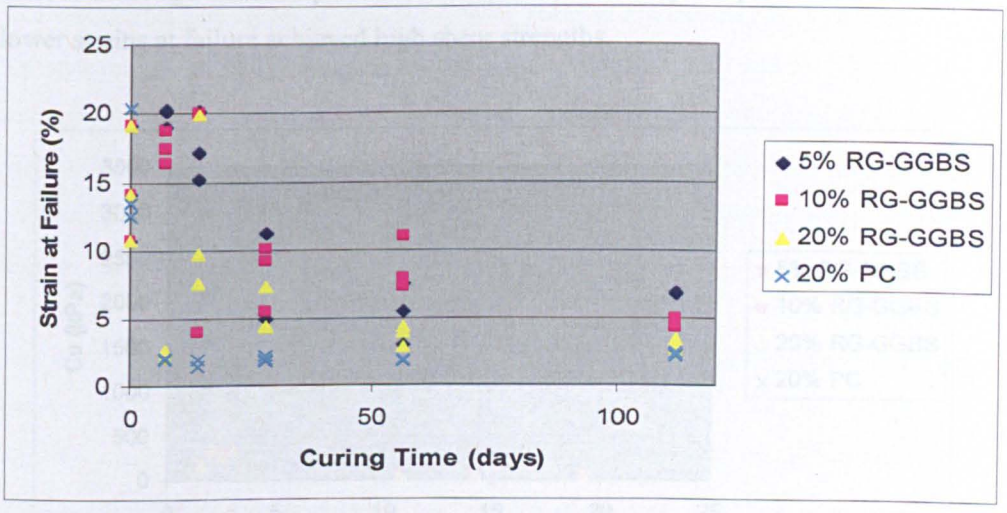



Figure 5.26, Soaked Glacial Till, strain at failure.

Table 5.11, Glacial Till average strains at failure.

| Sample | | Strain at Failure (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 15.6 | 19.8 | 17.53 | 9.2 | 5.33 | 6.9 |
| | un-soaked | | - | - | 1.55 | 2.08 | - |
| 10% (RG-GGBS) | soaked | 15.6 | 17.53 | 14.62 | 8.28 | 8.77 | 4.6 |
| | un-soaked | | - | - | 3.1 | 2.58 | - |
| 20% (RG-GGBS) | soaked | 15.6 | 16.7 | 12.43 | 5.33 | 3.69 | 2.89 |
| | un-soaked | | - | - | 1.89 | 2.41 | - |
| 20% (OPC) | soaked | 15.6 | 2.06 | 1.75 | 2.02 | 1.92 | 2.35 |
| | un-soaked | | 2.25 | 2.6 | 2.23 | 1.88 | 3.26 |

- no test conducted

Figures 5.27 and 5.28 Show the relationship between strain at failure and shear strength of the Glacial Till samples. As with the silty sand and London Clay samples there is a strong relationship between strain at failure. Only samples which exhibited lower strains at failure achieved high shear strengths.

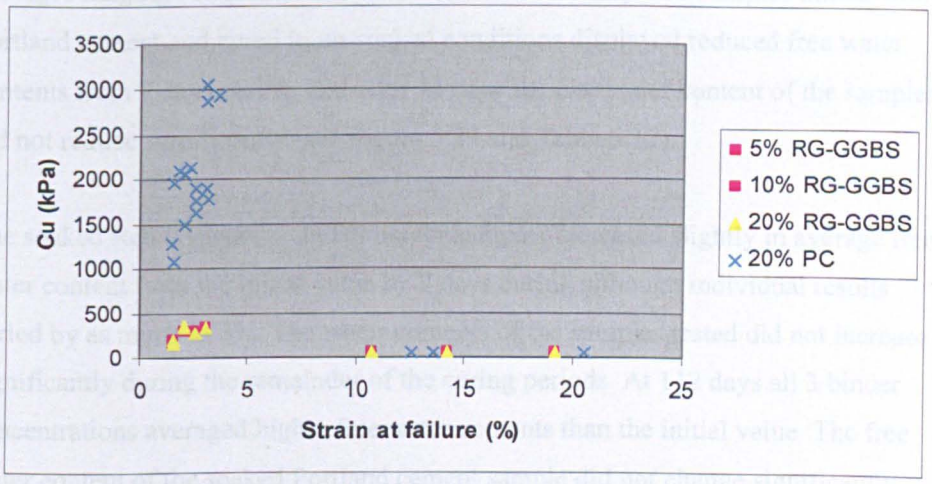



Figure 5.27 Strain at failure vs shear strength of un-soaked Glacial Till samples.

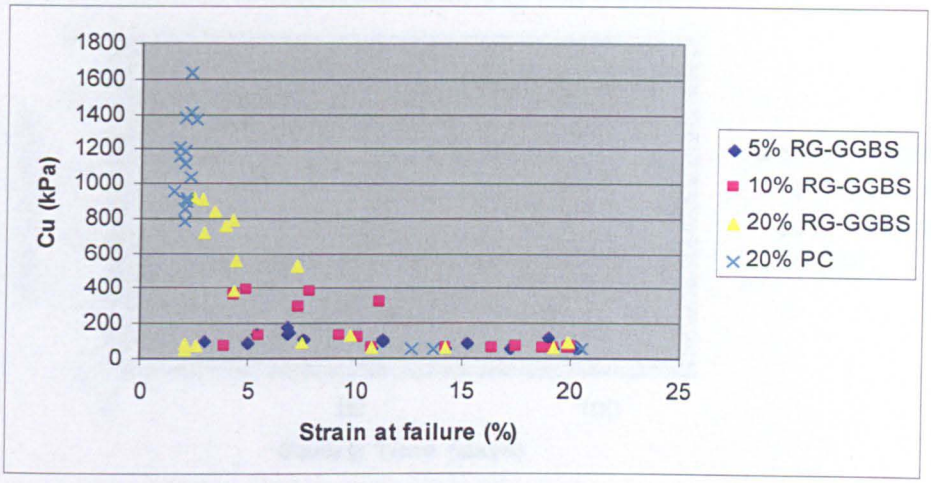



Figure 5.28 Strain at failure vs shear strength of soaked Glacial Till samples.

### 5.1.3.4 Water Content

The un-soaked Red Gypsum – GGBS binder samples did not display significantly reduced free water contents when the samples were tested at 28 and 56 days, the averages ranging between 20 and 21.5% (see Table 5.12). The samples mixed with Portland cement and cured in un-soaked conditions displayed reduced free water contents from 7 days curing, and after 14 days the free water content of the samples did not reduce significantly (see Figure 5.29 and Table 5.12).

The soaked Red Gypsum – GGBS binder samples increased slightly in average free water content from the initial value by 7 days curing although individual results varied by as much as 3%. The water contents of the samples tested did not increase significantly during the remainder of the curing periods. At 112 days all 3 binder concentrations averaged higher free water contents than the initial value. The free water content of the soaked Portland cement sample did not change significantly during the range of curing periods tested (see Figure 5.30). At 112 days the average free water content of the soaked Portland cement samples was 20.5% (Table 5.12).

Plots of water content against shear strength are included in Appendix C, these show that in both soaked and un-soaked conditions samples with lower water contents generally achieved higher strengths.

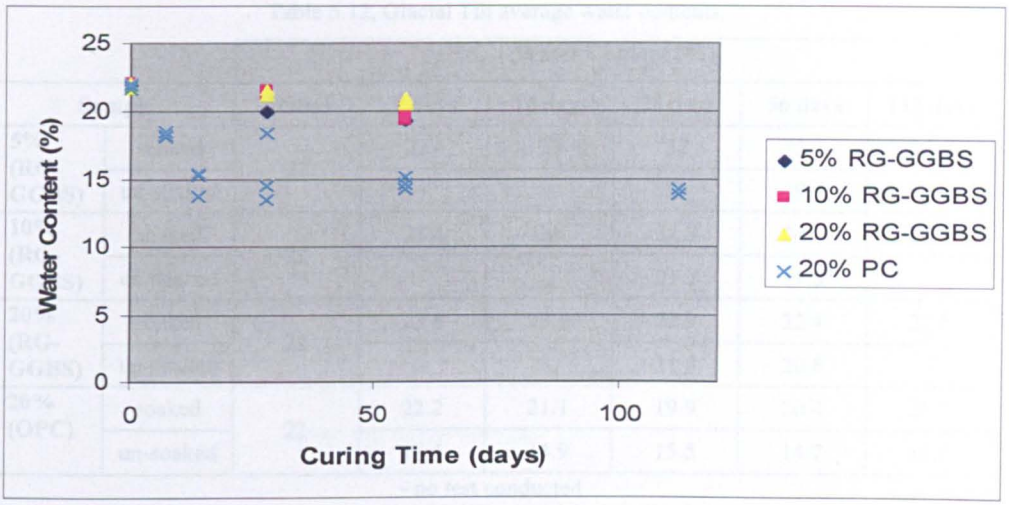



Figure 5.29, Un-soaked Glacial Till, water content.

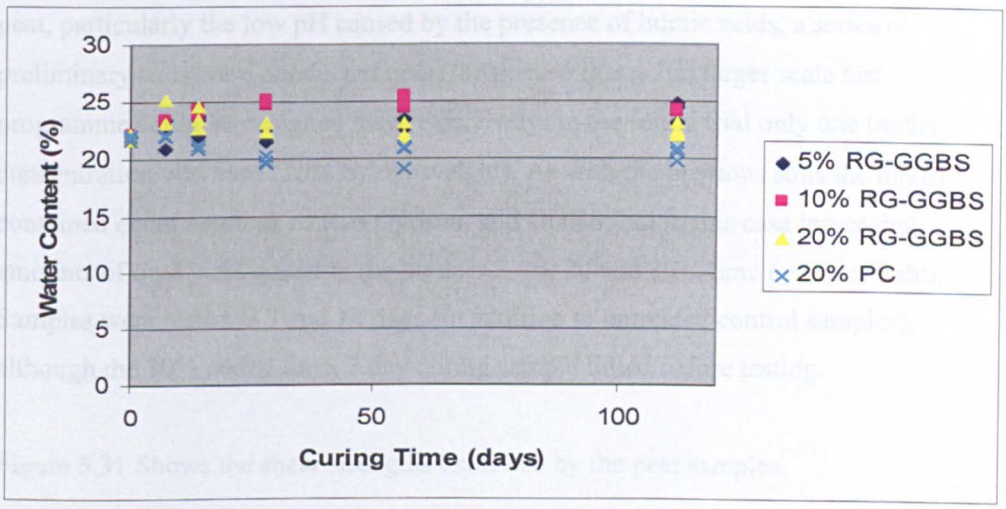



Figure 5.30, Soaked Glacial Till, water content.

Table 5.12, Glacial Till average water contents.

| Sample | | Water Content (%) | | | | | |
|---|---|---|---|---|---|---|---|
| | | Initial | 7 days | 14 days | 28 days | 56 days | 112 days |
| 5% (RG-GGBS) | soaked | 22 | 22.4 | 23 | 22 | 23.1 | 24.1 |
| | un-soaked | | - | - | 20 | 19.4 | - |
| 10% (RG-GGBS) | soaked | 22 | 23.4 | 24 | 24.9 | 24.9 | 24.3 |
| | un-soaked | | - | - | 21.2 | 19.8 | - |
| 20% (RG-GGBS) | soaked | 22 | 23.8 | 23.6 | 22.9 | 22.4 | 22.5 |
| | un-soaked | | - | - | 21.5 | 20.8 | - |
| 20% (OPC) | soaked | 22 | 22.2 | 21.1 | 19.9 | 20.4 | 20.5 |
| | un-soaked | | 18.4 | 14.9 | 15.5 | 14.7 | 14.1 |

- no test conducted

### 5.1.4 Irish Moss Peat

Because of the difficulties that are frequently experienced when attempting to solidify peat, particularly the low pH caused by the presence of humic acids, a series of preliminary tests were conducted upon the peat so that a 2nd larger scale test programme could be designed more effectively. In the initial trial only one binder concentration was used (20% by dry weight). As with the previous soils the binder contained equal amounts of Red Gypsum and GGBS, but in this case increasing amounts of lime were added to the binder (5, 10, 20 and 30% lime by dry weight). Samples were tested at 7 and 14 days (in addition to untreated control samples), although the 10% added lime, 7 day curing sample failed before testing.

Figure 5.31 Shows the shear strengths exhibited by the peat samples.

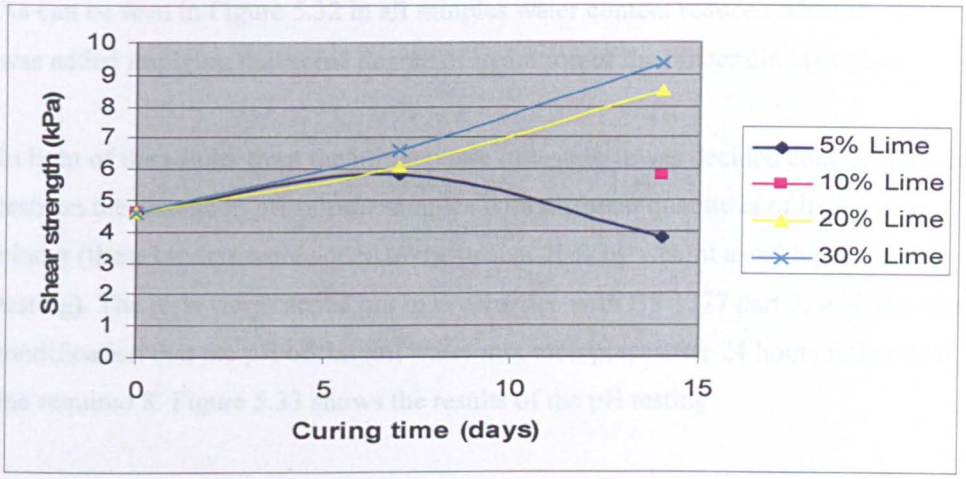



Figure 5.31 Initial peat mixing trial, shear strength results.

As can be seen in Figure 5.31 the 5 and 10% added lime samples did not exhibit significant strength gain, the 20 and 30% increased in strength over the 14 day curing period, but only from 4.5 kPa to 8.5 and 9.5kPa respectively, this is still very low. The same pattern also occurs in the Young's Modulus of the samples, whilst there was no significant change in the strains at failure of the samples over the curing period (see results in Appendix C).

Figure 5.32 Shows the free water content of the peat samples after testing .

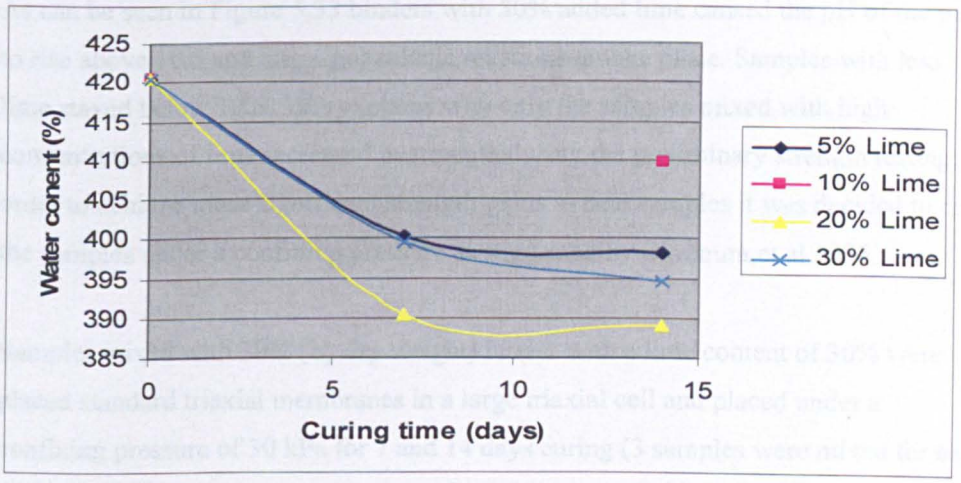



Figure 5.32, Initial peat mixing trial, post strength testing water contents.

As can be seen in Figure 5.32 in all samples water content reduced when the binder was added implying that some degree of hydration of the binder did take place.

In light of the results from the initial phase of testing it was decided conduct further tests on the change in pH of peat samples with different quantities of lime in the binder (these binders were added to the peat at 20% by weight as with previous testing). The tests were carried out in accordance with BS 1377 part 3, with the slight modification that the pH of the soil water mix took place after 24 hours rather than the required 8. Figure 5.33 shows the results of the pH testing.

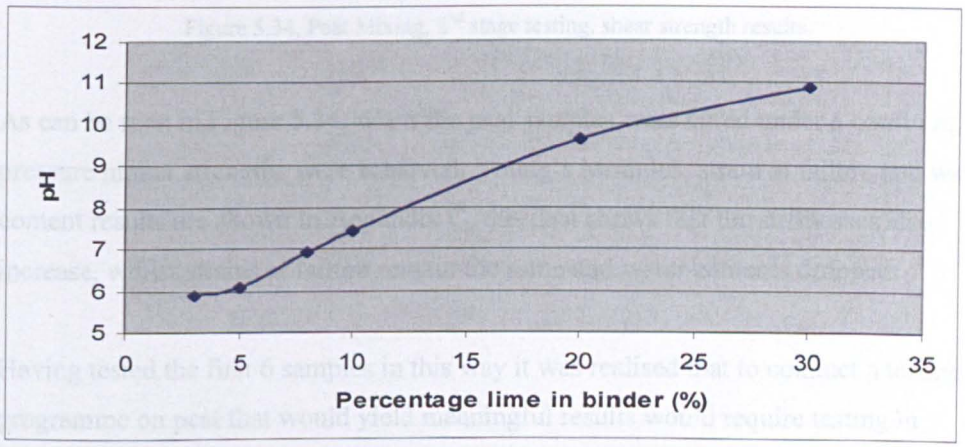



Figure 5.33, Peat mixing trial, pH results.

As can be seen in Figure 5.33 binders with 30% added lime caused the pH of the peat to rise above 10.5 and allow pozzolanic reactions to take place. Samples with less lime stayed below 10.5. This explains why only the samples mixed with high concentrations of lime increased in strength during the preliminary strength testing. In order to achieve more significant strength gains in peat samples it was decided to cure the samples under a confining pressure as suggested by Okumura et al 1996.

Samples mixed with 20% (by dry weight) binder with a lime content of 30% were placed standard triaxial membranes in a large triaxial cell and placed under a confining pressure of 30 kPa for 7 and 14 days curing (3 samples were mixed for each curing period). Figure 5.34 shows the shear strengths of the samples when tested.

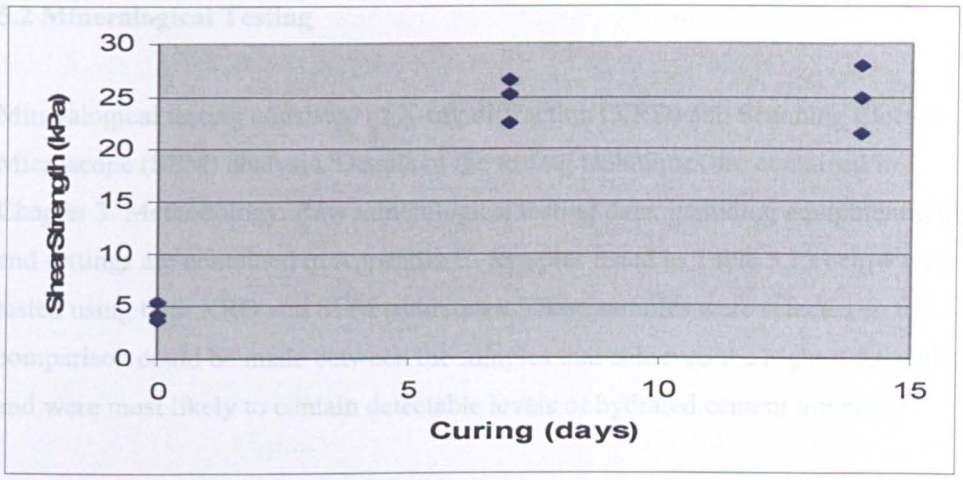



Figure 5.34, Peat Mixing, 2nd stage testing, shear strength results.

As can be seen in Figure 5.34, when the peat samples were cured under a confining pressure higher strengths were achieved. Young's Modulus, strain at failure and water content results are shown in Appendix C, this data shows that the stiffnesses also increase, whilst strains at failure remain the same and water contents dropped.

Having tested the first 6 samples in this way it was realised that to conduct a testing programme on peat that would yield meaningful results would require testing in excess of 100 samples. Curing these samples under a confining pressure and the increased care required when handling samples would be time consuming in the extreme, there was only the capacity for curing 3 samples at a time. In light of this and the low strength increases achieved it was decided to abandon the peat mixing so that research could concentrate on more promising areas. A separate study should be conducted on peat samples at a later date; further details will be included in the discussion section of this chapter.

## 5.2 Mineralogical Testing

Mineralogical testing consisted of X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis. Details of the testing techniques are contained in Chapter 3. Methodology. Raw mineralogical testing data, including equipment types and settings are contained in Appendix C. Samples listed in Table 5.13 below were tested using both XRD and SEM techniques. These samples were selected so that comparison could be made between the samples that achieved the highest strengths and were most likely to contain detectable levels of hydrated cement minerals.

Table 5.13, List of soil samples tested.

| | |
|---|---|
| Silty SAND | 20% RG-GGBS, Un-soaked |
| | 20% RG-GGBS, Soaked |
| | 20% Portland Cement, Un-soaked |
| | 20% Portland Cement, Soaked |
| London Clay | 20% RG-GGBS, Un-soaked |
| | 20% RG-GGBS, Soaked |
| | 20% Portland Cement, Un-soaked |
| | 20% Portland Cement, Soaked |
| Glacial Till | 20% RG-GGBS, Un-soaked |
| | 20% RG-GGBS, Soaked |
| | 20% Portland Cement, Un-soaked |
| | 20% Portland Cement, Soaked |

Mineralogical analysis was not conducted on peat samples due to the time and expense involved, instead it was felt it would be prudent to focus resources on soils which had undergone a more comprehensive range of physical testing.

### 5.2.1 X-ray Diffraction Analysis

Full copies of the XRD data is contained in Appendix C, the results are summarised in Table 5.14.

Table 5.14, Summary of Soil Mixing X-ray diffraction results.

| | Red Gypsum – GGBS | | Portland Cement | |
|---|---|---|---|---|
| | **Un-soaked Curing** | **Soaked Curing** | **Un-soaked Curing** | **Soaked Curing** |
| **Silty Sand** | Kaolinite<br>Gypsum<br>Ettringite<br>Quartz | Kaolinite<br>Gypsum<br>Ettringite<br>Quartz | Kaolinite<br>Portlandite<br>Calcite<br>Quartz | Kaolinite<br>Portlandite<br>Calcite<br>Quartz |
| **London Clay** | Gypsum<br>Ettringite<br>Quartz<br>Muscovite | No Sample | Kaolinite<br>Calcite<br>Ettringite<br>Quartz<br>Muscovite | Ettringite<br>Quartz<br>Muscovite |
| **Glacial Till** | Kaolinite<br>Gypsum<br>Quartz | Kaolinite<br>Gypsum<br>Ettringite<br>Quartz | Kaolinite<br>Portlandite<br>Calcite<br>Quartz | Kaolinite<br>Portlandite<br>Calcite<br>Quartz |

### 5.2.2 Scanning Electron Microscope Analysis (SEM)

Polished thin sections were analysed at 100 and 1000 times magnification. A number of the images recorded are reproduced in this section. Full size copies of all the images recorded are contained in Appendix C.

#### 5.2.2.1 Silty Sand

Figures 5.35 and 5.36 show that little difference can be observed between the sample with Portland cement binder and the one with Red Gypsum –GGBS binder. The main observable difference is the presence of GGBS in Figure 5.35 and Portland cement in Figure 5.36.

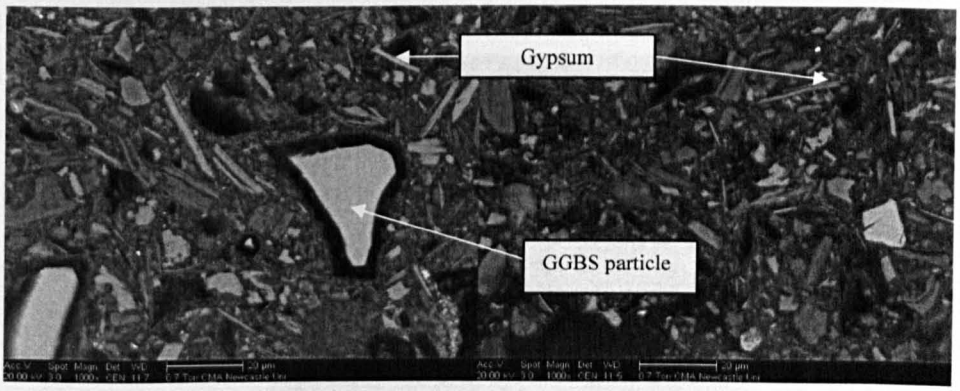



Figure 5.35, SEM images of Silty sand mixed with 20% RG-GGBS binder (left, un-soaked curing, right, soaked curing).

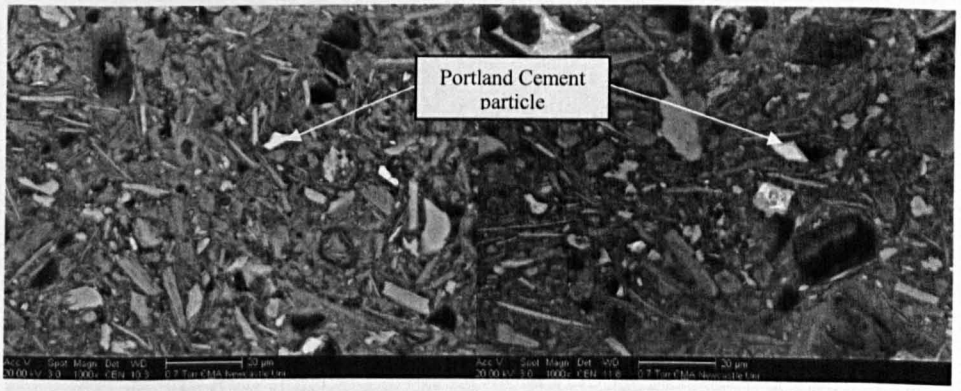



Figure 5.36, SEM images of Silty sand mixed with 20% Portland Cement binder (left, un-soaked curing, right, soaked curing).

There are many needle shaped crystals in all the images, in the case of the Red Gypsum – GGBS samples these are gypsum, ettringite and some clay minerals (probably Kaolinite). In the Portland Cement samples the needle shaped crystals are calcite and clay minerals, XRD testing having shown no evidence of ettringite. There appears to be no difference between samples cured in soaked and un-soaked conditions.

### 5.2.2.2 London Clay

As stated previously, it was not possible to analyse a sample of London Clay mixed with 20% Red Gypsum – GGBS binder and cured for 56 days in soaked conditions, (due to the accidental disposal of a number of samples whilst the author was on holiday) so it is not possible to compare samples of different curing conditions. Figures 3.37 and 3.38 show SEM images of the London Clay samples.

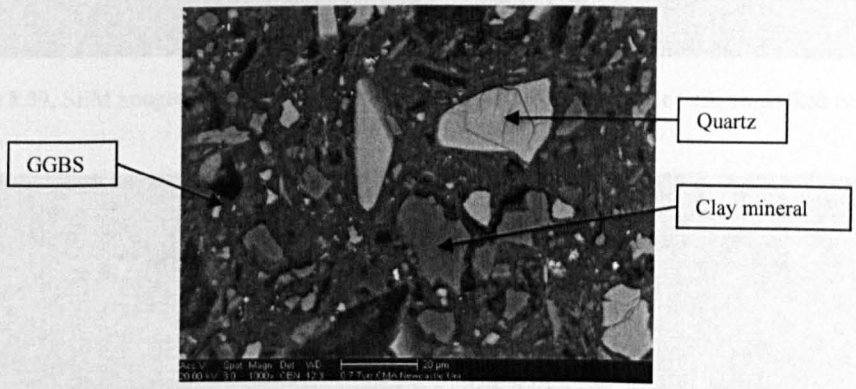



Figure 5.37, SEM image of London Clay mixed with 20% RG-GGBS binder, un-soaked curing.

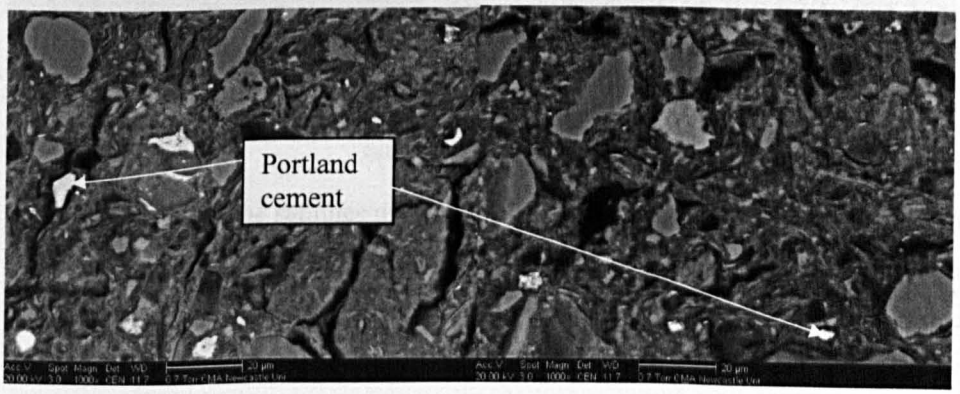



Figure 5.38, SEM images of London Clay mixed with 20% Portland Cement binder (left, un-soaked curing, right, soaked curing).

In the case of the Portland cement samples, little difference can be seen between the two samples. There also appears to be little difference between the samples mixed with Red Gypsum and those mixed with Portland cement.

### 5.2.2.3 Glacial Till

From Figures 5.39 and 5.40 it can be seen that there is no observable difference between samples with the same binders but cured under different conditions.

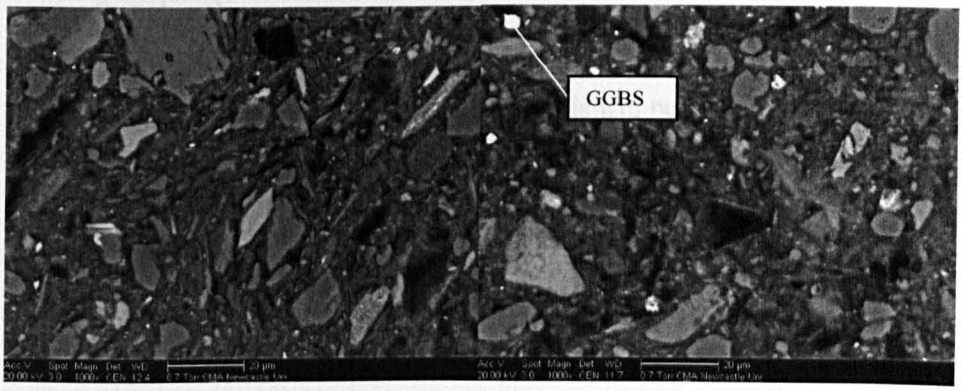



Figure 5.39, SEM images of Glacial Till mixed with 20% RG-GGBS binder (left, un-soaked curing, right, soaked curing).

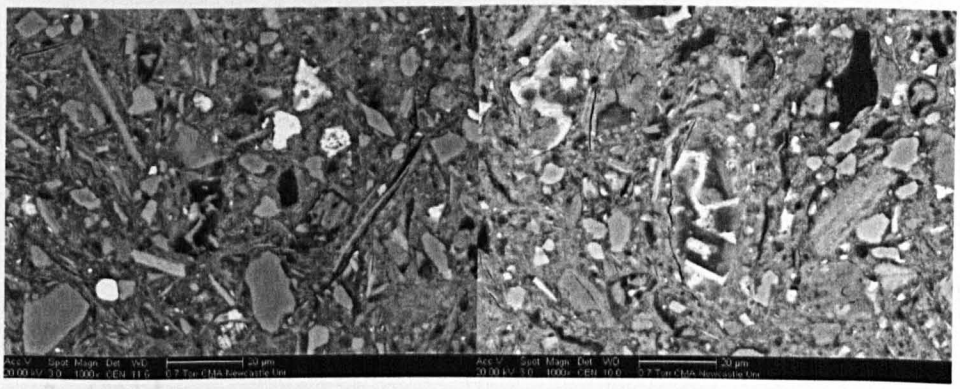


Figure 5.40, SEM images of Glacial Till mixed with 20% Portland Cement binder (left, un-soaked curing, right, soaked curing).

It is noticeable that the samples mixed with Portland Cement have significantly more needle shaped crystals. Some are present in the Red Gypsum – GGBS samples, these these gypsum, ettringite and some clay minerals (probably Kaolinite). In the Portland cement samples the needle shaped crystals are calcite and clay minerals, as no evidence of ettringite or gypsum was indicated by the XRD analysis.

### 5.3 Soil Mixing Discussion

Four main themes arise from the results of the soil mix testing, which are:

- Strength/stiffness development and strain behaviour
- Water content
- Mineralogy
- Sample pH

Each of these elements are interdependent and this will be explained in the following sections. Sections 5.3.1 to 5.3.4 discuss the different themes in detail, which are then summarised and concluded in section 5.4

#### 5.3.1 Strength/stiffness development and strain behaviour

As has been shown earlier in this chapter the rate and magnitude of strength development observed in the tested soils when mixed with binders differed widely, Figures 5.41 and 5.42 illustrate this. The strength/stiffness and strain at failure of each of the soils is discussed here separately.

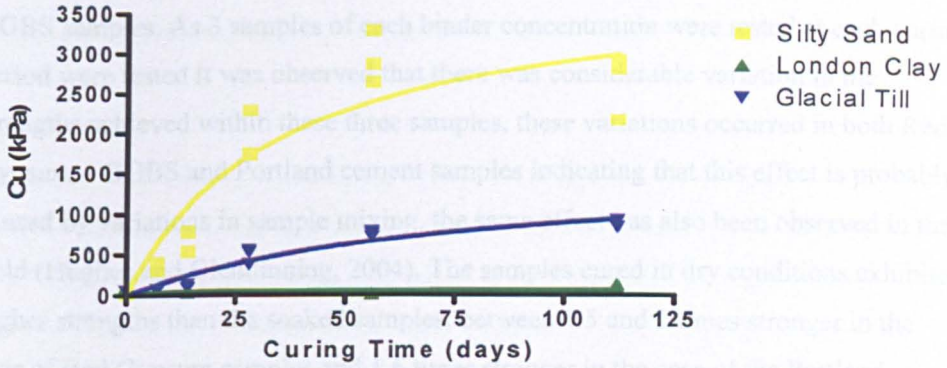



Figure 5.41, Comparison of 20% soaked curing soil mixing shear strengths.

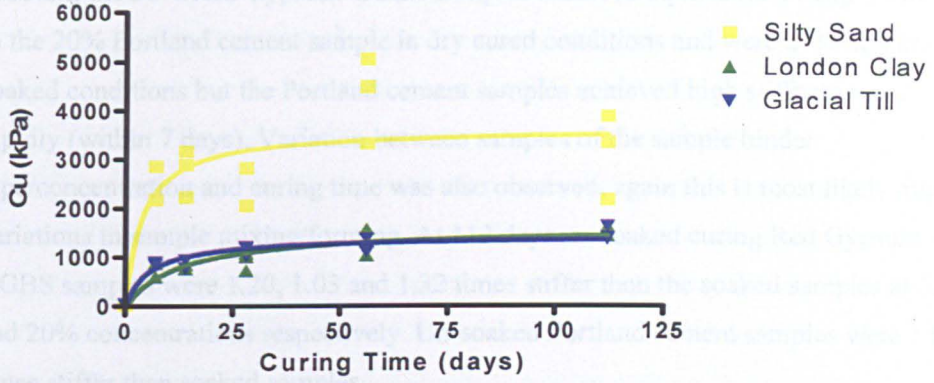



Figure 5.42, Comparison of 20% un-soaked curing soil mixing shear strengths.

**Silty Sand**

In the silty sand samples higher concentrations of Red Gypsum-GGBS yielded higher shear strengths (see Figures 5.1 and 5.2), as would be expected, and demonstrates that it is the binder that is increasing the strength of the samples and not another factor (sample drying etc). The addition of 20% Red Gypsum-GGBS samples achieved an equivalent strength to the addition of 20% Portland cement over the longer curing periods in dry curing conditions and achieved 80% of the shear strengths produced by Portland cement in soaked curing conditions, indicating that over the curing periods tested the Red Gypsum – GGBS binder is an effective replacement for Portland cement when improving silty sand. It must be added that the Portland cement samples gained strength much more rapidly than the Red Gypsum – GGBS samples. As 3 samples of each binder concentration were tested at each curing period were tested it was observed that there was considerable variation in the strengths achieved within these three samples, these variations occurred in both Red Gypsum – GGBS and Portland cement samples indicating that this effect is probably caused by variations in sample mixing, the same effect has also been observed in the field (Hughes and Glendinning, 2004). The samples cured in dry conditions exhibited higher strengths than the soaked samples, between 1.5 and 2 times stronger in the case of Red Gypsum samples and 1.6 times stronger in the case of the Portland cement samples.

As could be expected the stiffness results mirrored the strength results (see Figures 5.3 and 5.4). Higher concentrations of Red Gypsum-GGBS yielded higher Young's

Modulii, the 20% Red Gypsum-GGBS samples achieved equivalent Young's Modulii to the 20% Portland cement sample in dry cured conditions and were 20% higher in soaked conditions but the Portland cement samples achieved high stiffness more rapidly (within 7 days). Variation between samples of the sample binder type/concentration and curing time was also observed, again this is most likely due to variations in sample mixing/forming. At 112 days un-soaked curing Red Gypsum - GGBS samples were 1.20, 1.03 and 1.32 times stiffer than the soaked samples at 5, 10 and 20% concentrations respectively. Un-soaked Portland cement samples were 1.80 times stiffer than soaked samples.

10 and 20% Red Gypsum -GGBS concentration samples exhibited low (<4.3%) strains at failure at 7 days curing stayed below 5% for the range of curing periods tested (see Figures 5.5 and 5.6). The 5% concentration samples averaged higher (approx 14%) strains at failure until 28 days curing when they to dropped below 5%. This is probably due to the higher strengths achieved by the larger binder concentrations making the samples more brittle in nature. The Portland cement samples exhibited approximately the same strains at failure as the 20% Red Gypsum-GGBS binder samples. Un-soaked samples displayed low strains at failure earlier but all samples exhibited similar strains at failure at 112 days, in untreated soils higher water contents cause samples to behave in a more plastic manner (until the liquid limit is reached). As with the strength and stiffness there was considerable variation between samples of the same binder type/concentration and curing time most likely caused by variations in mixing and sample formation.

**London Clay**

Unlike the Silty Sand samples the dry cured Red Gypsum -GGBS London Clay samples reduced in shear strength compared to untreated sample (See Figures 5.11 and 5.12). Although by 56 days all 3 binder concentrations were greater then 1.5 times stronger than the initial sample (although the 112 day samples achieved lower strengths than the untreated samples). No Red Gypsum -GGBS concentrations were significantly stronger than others. Although there was an increase in strength it was not very significant and the fact that larger additions of binder did not yield larger increases in strength indicates that the strength increase may be in whole or in part due to another factor (i.e. drying). By contrast the Portland cement samples cured in

dry conditions increased in strength significantly (in excess of 500kPa) by 7 days and achieved significantly higher strengths than the Red Gypsum -GGBS samples.

The soaked Red Gypsum -GGBS samples reduced in strength compared to the initial untreated samples, and by 56 days all 3 concentrations averaged shear strengths of below 20kPa (approx 20% of the original strength). Only the 20% Red Gypsum - GGBS concentration samples were stronger at 112 days than the initial untreated samples and only by 20kPa. The reduction in strength is almost certainly due to an increase in water contents of about 10% as the sample soaked. Given that the samples did begin to increase in strength slightly after 56 days indicates that the binder may have had some effect but it was not enough to counter the effect of the wetting up of the samples. As with the dry cured samples the Portland cement mixes increased in strength significantly (approx 700kPa) by 7 days and were significantly stronger than the Red Gypsum -GGBS (10 times stronger) samples throughout the range of curing periods. Un-soaked Red Gypsum-GGBS samples were marginally stronger than the soaked ones the un-soaked Portland cement samples also achieved higher shear strengths than the soaked samples.

All concentrations of RG-GGBS exhibited reduced Young's Moduli compared to the initial untreated value by 7 days, values only increased to over the initial value at the 56 day test (see Figures 5.13 and 5.14). At 112 days values of Young's Modulus were not significantly greater than the initial value. Un-soaked Portland cement samples achieved significantly higher Young's Modulus than Red Gypsum-GGBS.

All concentrations of the soaked Red Gypsum-GGBS exhibited reduced Young's Modulus after 7 days. Only the 20% RG-GGBS samples achieved higher Young's Modulus than the initial untreated samples at 112 days curing. Soaked Portland cement achieved significantly higher Young's Moduli than the initial untreated samples and the Red Gypsum -GGBS samples. Un-soaked Red Gypsum -GGBS samples achieved marginally higher Young's Modulus than the soaked samples. Un-soaked Portland cement samples achieved higher Young's Modulus than the soaked samples. These results mirror the shear strength results as would be expected.

Un-soaked Red Gypsum -GGBS samples did not display significant changes in strains at failure compared to the initial untreated samples, although samples with higher binder concentrations displayed marginally lower strains at failure than those with lower binder concentrations (see Figures 5.15 and 5.16). This is consistent with the shear strength and Young's Modulus results, as there was only very limited improvement in the strength and stiffness in the samples they would also behave in a similar plastic manner to the untreated samples. The un-soaked Portland cement samples displayed significantly lower strains at failure than the Red Gypsum -GGBS samples, consistent with the samples stiffening and becoming more brittle.

Soaked Red Gypsum -GGBS samples exhibited higher strains at failure than the initial untreated samples. The water content increase due to being soaked would have caused the soil to fail in a more plastic way with no hardening of the sample to making it more brittle.

As with the un-soaked samples the soaked Portland cement samples exhibited significantly lower strains at failure than the RG-GGBS samples.

**Glacial Till**

The unsoaked Glacial Till Red Gypsum-GGBS samples achieved approximately 5 times the strength of the untreated samples after 56 days curing (see Figures 5.21 and 5.22). However, no significant difference in strength was observed between samples of different binder concentrations at either 28 or 56 days curing (unfortunately these were the only curing periods tested). This implies that increases in binder percentage does not increase the effect on shear strength and that another factor may have been involved in the strengthening of the samples. The un-soaked Portland cement sample achieved significantly higher shear strengths than the Red Gypsum-GGBS samples. These high strengths developed within the first 7 days of curing but showed significant variations over the course of the curing periods tested this is probably due natural variation in the till samples.

The soaked Red Gypsum-GGBS samples showed only slight increases in strength by 14 days curing. The 20% binder addition samples began to show significant strength gain first at 28 days curing (9 times stronger than the initial samples), the 10%

samples displayed strength gains at 56 days (6 times stronger than initial samples). The 5 % binder addition samples gained strength slowly throughout the range of curing periods but were only 2.5 times stronger than the initial samples at 112 days. In addition to strengthening faster, samples with higher binder concentrations attained higher strength over the full range of curing periods. This contrasts with the un-soaked samples and indicates that hydration of the Red Gypsum – GGBS binder is the main factor strengthening the soil. The soaked Portland cement samples achieved significantly higher shear strengths than the Red Gypsum-GGBS samples, the most rapid gain in strength occurring between 0 and 7 days but the strength continued to increase through to 112 days curing. The soaked Red Gypsum-GGBS samples achieved higher strengths than the un-soaked samples. Un-soaked Portland cement samples achieved higher shear strengths than soaked samples this also indicates that the dry curing conditions prevented the Red Gypsum – GGBS binder samples from hardening.

As with the shear strength results the un-soaked Red Gypsum-GGBS samples achieved 5 times the Young's Moduli of the initial samples but there was no evidence that greater Red Gypsum-GGBS binder concentrations cause greater Young's Moduli (see Figures 5.23 and 5.24). The un-soaked Portland cement samples achieved significantly higher Young's Moduli than the Red Gypsum-GGBS samples.

The Soaked Red Gypsum-GGBS samples doubled in Young's Modulus by 7 days, and it was clear that larger concentrations of Red Gypsum-GGBS binder caused greater increases in Young's Moduli. The soaked Portland cement samples achieved significantly higher Young's Moduli than the RG-GGBS samples. This is consistent with the shear strength results.

When tested after 28 and 56 days curing the dry cured Red Gypsum – GGBS samples exhibited strains at failure lower than 3.1%, significantly lower than the initial untreated samples but there was no indication that higher concentrations of Red Gypsum-GGBS binder caused lower strains at failure. This indicates that despite not exhibiting large increases in strength the samples did become brittle with time. The un-soaked Portland cement samples exhibited similar strains at failure to the Red

Gypsum-GGBS samples but as has already been discussed were significantly stronger.

Initially the soaked Red Gypsum-GGBS binder samples displayed increased strains at failure but these reduced to well below the values of the initial untreated samples by 28 days (see Figures 5.25 and 5.26). This can be explained by the samples taking in water initially and softening, before the binder then began to take effect and harden them. The samples with larger binder concentrations exhibited smaller strains at failure this is consistent with the shear strength and Young's Modulus results. The Portland cement samples exhibited lower strains at failure earlier than the Red Gypsum -GGBS samples.

**Irish Moss Peat**

As stated earlier in this chapter the nature of peaty soils, particularly the low pH, necessitated a different approach to testing the binder in this type of soil. The smaller testing programme that was conducted (before being suspended) showed that when samples mixed with Red Gypsum binder were cured in un-soaked, unconfined conditions the strengths achieved, although double the value of the untreated soil, were extremely low (less than 10 kPa) (see Figure 5.31). When the second phase of the testing was conducted higher strengths were achieved, although these were still less than 30 kPa (see Figure 5.34). As with the other soils tested the stiffness of the samples mirrored the shear strengths achieved with both the un-confined samples and confined samples each exhibiting increases in stiffness. For all samples the strains at failure remained essentially unchanged, averaging over 15% throughout the curing periods tested and not exhibiting the reductions seen in the other soils.

The modified strength properties achieved in the Peat samples were not sufficient to be desirable from an engineering standpoint. To put it simply the addition of binder caused a very soft soil to become very slightly less soft.

To summarise, the RG-GGBS binder was significantly effective compared to Portland cement when mixed with Silty Sand. The binder also achieved positive results when mixed with Glacial Till though was not as effective as Portland cement in this case. When mixed with London Clay and Irish Moss Peat the RG-GGBS

binder was not effective. Where the binder was effective samples being cured in soaked conditions did not prevent hardening although the magnitude of strength and stiffness gain was not as pronounced, this is discussed further in section 5.3.2.

Comparing the rate of hardening between the two binder types it is clear that Portland cement hardens much more rapidly than the Red Gypsum – GGBS binder. As will be discussed in section 5.4 this has implications for acceptability on site. The reasons for the different rate of hardening are due to the type of reaction occurring in the Red Gypsum binder samples (i.e. pozzolanic) this is discussed in section 5.3.3.

Across all the samples there was a strong link between the shear strength achieved and the strain at failure, samples which exhibited high strengths also exhibited low strains at failure. The engineering implications of this are that when these binders are used to improve soils a consequence will be that cracking may occur at high strains, this may be undesirable where strong vibration is likely to occur (i.e. earthquakes) but are unlikely to undergo creep.

### 5.3.2 Water Content

Figure 5.43 shows the relationship between shear strength and water content in un-soaked silty sand samples. As can be seen in Figure 5.40 there is a strong relationship between shear strength and the free water content of the samples (plots of this relationship in the other soils tested can be found in Appendix C). The reasons for the change in water content over the curing periods tested are not entirely the same for each of the soils tested. Each soil is discussed in this section separately.

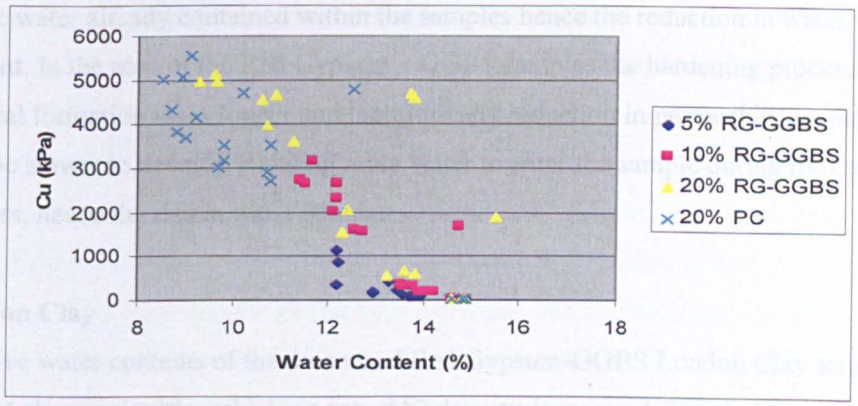



Figure 5.43 Shear strength/water content relationship in un-soaked silty sand.

**Silty Sand**

When cured in dry conditions all Red Gypsum-GGBS Silty Sand binder samples reduced in water content from the initial value. Greater concentrations of binder exhibited larger reductions in water content. This is consistent with the hydration reactions within the binder using the water within the soil. Where there is more binder more water is required for the reactions to take place. Portland cement samples reduced in water content more rapidly (less than 11% within 7 days) and displayed lower water contents at 112 days than the 20% RG-GGBS samples. This is consistent with the strength development of the samples, the Portland cement samples achieving high strengths within 7 days.

Soaked Silty Sand Red Gypsum -GGBS binder samples increased in water content by 7 days and remained higher than the initial untreated sample throughout the range of curing periods. Samples with higher binder concentrations displayed the highest increase in water content at 112 days. This indicates that whilst the binder would be using water in the hydration reactions more water was entering the samples throughout the range of curing periods. In contrast to the Red Gypsum – GGBS samples the Portland cement samples reduced in water content and remained lower than the initial un-treated samples throughout the range of curing periods tested. The reason for the difference in water content behaviour may be due to the faster reaction of the Portland cement binder. As the majority of the Portland cement reactions take place within 7 days this will form hydrated cement minerals in the pore spaces of the samples very quickly reducing the permeability of the sample thus preventing further water from entering the sample. The only source of water for the reactions would then be the water already contained within the samples hence the reduction in water content. In the case of the Red Gypsum – GGBS samples the hardening process and mineral formation takes longer and therefore any reduction in permeability would also be slower to develop allowing more water to enter the sample during the curing process, hence the rise in water content.

**London Clay**

The free water contents of the un-soaked Red Gypsum-GGBS London Clay samples did not change significantly during the 112 days curing period. This indicates that no water was used in hydration reactions of the binder. The free water content of the

Portland cement samples reduced by more than 6% over the 112 day curing period consistent with water being used in the hydration of the cement particles. As with the silty sand samples the water content reduced rapidly between 7 days then levelled off, again being consistent with the formation of hydrated cement minerals causing the permeability of the samples to be reduced and thus restricting the ingress of water into the sample.

The free water contents of the Red Gypsum -GGBS samples increased by less than 5% in the 112 day curing samples consistent with the strength and stiffness loss and the plastic failure behaviour. The soaked Portland cement samples did exhibit an increase in free water content but this increase was not as much as the Red Gypsum – GGBS samples. The increase took place during the first 14 day after which the water content was effectively unchanged. The increase and then levelling off is again believed to be due to the formation of hydrated cement minerals early in the curing period causing the permeability of the samples to decrease restricting further inflow of water.

**Glacial Till**

The soaked Red Gypsum-GGBS Glacial Till samples did not exhibit significant reductions in free water content by 56 days and the samples with greater concentrations of Red Gypsum-GGBS binder did not exhibit significantly lower free water content. This means that the lack of reaction of the binder and only slight hardening of the samples discussed previously cannot be accounted for by drying of the samples and that some other factor prevented the samples from gaining significant strength. By contrast the Portland cement samples exhibited a reduction in free water content to 15% by 14 days. The free water content of the un-soaked Portland cement samples did not change significantly after this.

The soaked Red Gypsum-GGBS samples exhibited slightly raised free water contents throughout the range of curing periods indicating that water was able to enter the samples and was not all used by the reaction of the binder. The soaked Portland cement samples exhibited slightly lower free water contents than the Red Gypsum-GGBS samples and the initial untreated samples.

**Irish Moss Peat**

In the first phase of the Peat testing there was only a slight reduction in water content from 420% to between 390 and 410% for the different binder concentrations. As a percentage of the overall water content this is not a significant reduction. By contrast the samples cured in confined conditions reduced in water content from 410% to 200%, a drop of more than half. As discussed previously in this chapter the samples cured in confined conditions achieved higher strengths it is most likely that the increased reduction in water content seen in these samples when compared to the un-confined ones is at least in part due to a greater degree of hydration/formation of hydrated cement minerals occurring.

### 5.3.3 Mineralogy

As previously stated the mineralogical analysis performed upon the soil mix samples was conducted using XRD and SEM techniques. Whilst XRD is a powerful technique for identifying minerals, the technique used cannot identify amorphous minerals or quantify the amounts of any minerals identified. It was hoped that backing up the XRD analysis with SEM work would address this deficiency.

The X-ray diffraction analysis indicated the presence of kaolinite, gypsum, ettringite and quartz in the soaked and un-soaked Red Gypsum – GGBS silty sand samples tested (20% binder concentration). The presence of Kaolinite and Quartz can be explained by the fact that they are the major components of the silty sand, the gypsum detected is a component of the binder. The presence of the Ettringite (indicative of pozzolanic reactions), also observed in the binder only samples tested, within the samples is a possible cause for concern although during the testing programme no swelling of the samples was observed. Also it should be noted that whilst the X-ray diffraction analysis technique used can identify the presence of ettringite it cannot quantify it and only small amounts may have formed. As with the Red Gypsum – GGBS samples X-ray analysis of the Portland cement silty sand samples indicated the presence of quartz and kaolinite. In addition portlandite and calcite were also present, the Calcite is a component of the Portland cement whereas the portlandite is a product of the hydration of cement. Neither of the Portland cement samples exhibited the presence of ettringite.

X-ray analysis of a sample of London Clay with a 20% addition of Red Gypsum – GGBS binder cured for 56 days indicated the presence of gypsum, ettringite, quartz and muscovite (see Table 5.14). The quartz and muscovite are almost certainly constituents of the original soil, the quartz as sand grains and muscovite being a common mineral found in igneous rocks which often occur in gravels in clay soils. As with the silty sand samples tested the gypsum is undoubtedly the gypsum used in the binder and ettringite has already been seen to form in the Red Gypsum – GGBS binder. Unfortunately it was not possible to test a sample of London Clay with a 20% addition of Red Gypsum – GGBS cured in soaked conditions to make a comparison.

The Portland cement London Clay samples both indicated the presence of ettringite, quartz and muscovite (the dry cured sample additionally indicated the presence of kaolinite and calcite). Again the quartz and muscovite are easily explained as constituents of the original soil but it is interesting to note the presence of ettringite in the samples, especially as no gypsum was added. This indicates that there were sulphates already in the soil prior to mixing. Subsequent analytical testing showed that sulphate was present within the untreated London Clay samples (see Results contained in Appendix A). This could be the reason why the Red Gypsum – GGBS samples did not exhibit significant strength gains; oxidation of the sulphates within the soil would cause the production of sulphuric acids in turn causing the pH of the soil to drop. If the pH of the soil is less than 10.5 pozzolanic reactions cannot take place. The kaolinite and calcite detected in the dry cured samples are not significant, as previously mentioned kaolinite is a common clay mineral and calcite is present in Portland cement.

X-ray analysis of the Red Gypsum – GGBS Glacial Till samples indicated the presence of kaolinite, gypsum and quartz (see Table 5.14) in both soaked and un-soaked samples; additionally ettringite was detected in the soaked sample. The fact that ettringite was present in the soaked sample and not the un-soaked one combined with the results of the physical tests indicates that for some reason the binder in the unsoaked samples did not react. X-ray analysis of the Portland cement samples indicated the presence of portlandite, calcite, kaolinite and quartz as discussed in previous sections, portlandite is a product of the hydration of cement and calcite is a

present in portlandite and therefore probably represents un-hydrated cement in the samples.

Unfortunately the results from the SEM analysis were not as conclusive as the XRD analysis. It was hoped that the SEM analysis may indicate the presence of C-S-H or C-A-H, unfortunately no conclusive proof of either mineral was found. There are several reasons why the technique proved unsuccessful in this case. Firstly, the samples were composed of soil mixed with only 20% by dry weight of binder making hydrated cement mineral more difficult to find. Secondly, in order to make use of the elemental analysis facility on the SEM polished sections were used rather than broken sections, and this meant it was more difficult to identify minerals by their morphology. Finally, the elemental analysis system used was not (and could not be) calibrated to detect hydrogen. This made identifying any hydrated minerals more difficult.

### 5.3.4 pH

The strength development of the Red Gypsum binder is dependant upon Pozzolanic reactions taking place. As stated previously this is dependant upon a pH of at least 10.5 to bring alkali silica into solution. Where significant strength development has not occurred in the samples it can in each case be linked back to low pHs within the soil, which is in turn linked to the initial mineral composition of the soil being treated. The addition of lime has, in the case of the silty sand and soaked Glacial Till, been sufficient to increase the pH to the required level. In the case of the un-soaked Glacial Till samples increasing the lime content of the binder by 1% would almost certainly be sufficient to increase the pH by the required amount. The original tests to assess the quantity of lime required to increase the pH to the sufficient value was conducted in accordance with BS 1377 (BSI, 1990). In this test the pH of the soil water solution is measured after a minimum of 8 hours but the reactions within the soil that reduced the pH through the generation of acids took place over a longer period. This meant that whilst the calculated quantities of lime were sufficient to boost the pH in the short term they were not necessarily sufficient to maintain those levels for the longer term required for Pozzolanic reactions to take place.

### 5.3.5 General Discussion, Conclusions and Recommendations

Many construction specifications require strengths to be achieved by 28 days (Hanson et al, 2001), however faster hardening is desirable as it facilitates rapid construction. This would make Red Gypsum – GGBS binders less desirable compared to Portland cements as the results clearly demonstrate that the Portland cement samples developed high strengths much more rapidly than the Red Gypsum-GGBS samples.

The speed of the strength development was similar in all of the soils tested and there was no difference to the rate of hardening when samples were cured in soaked or un-soaked conditions. The magnitude of the strengths differed however, in the case of the silty sand Red Gypsum binders achieved similar strengths and stiffnesses to those achieved by the Portland cement binders over the curing periods tested and results indicate that were samples to be tested over longer curing periods they would achieve greater strengths and stiffnesses than samples mixed with Portland cement. In the case of London Clay samples the strength and stiffness of the Red Gypsum-GGBS samples was not significantly greater than the initial untreated samples and were significantly lower than those treated with Portland cement. Whilst in the case of the Glacial Till samples RG-GGBS samples there was a significant increase in the strength and stiffness of the RG-GGBS samples over the initial untreated samples but this was not as pronounced as achieved by the Portland cement samples. In the case of the un-soaked RG-GGBS Glacial Till samples the degree of strength increase was lower implying that there was an error when the samples were mixed with not enough lime being added to fully activate the binder. The peat samples achieved very little strength and stiffness increase in either un-confined or confined curing conditions. The strain at failure results from all the soils show a strong relationship with maximum strengths attained in that where samples exhibit high strengths they also exhibit low strains at failure, this applies equally to the Portland cement samples as it does to the RG-GGBS samples.

The obvious conclusion from this is that the effectiveness of the binder is strongly effected by the type of soil it is mixed with. The results indicate that there are two main factors that contribute to this effect. These are the mineralogy of the soil and its effect on the pH, and the structure of the soil.

The mineralogy of the soil effects the activation of the binder though it effect on the pH of the pore water necessary for the hydration of the Red Gypsum-GGBS binder. As discussed previously the reactions that causes the strength and stiffness gain in the Red Gypsum-GGBS binder is pozzolanic and as such requires the pH of the water to be in excess of 10.5. In the case of the Silty Sand the mineralogy is such that it has little effect on the pH of the water added during mixing therefore the lime added as part of the binder caused the pH to increase well in excess of 10.5 thus permitting pozzolanic reactions to take place. In the Glacial Till the mineralogy was such that it did cause the pH of the samples to drop, in the un-soaked samples the lime added was insufficient to maintain pHs above 10.5 and therefore the binder did not activate fully. In the soaked samples more lime was added and the samples achieved higher pHs and therefore a more pronounced strength gain was achieved. In the London Clay samples significant quantities of sulphate was present, when water was added the sulphate oxidised to sulphate and sulphuric acid was generated drastically reducing the pH and preventing the Red Gypsum-GGBS binder from going off. In the peat samples the acid generated was humic, produced by a reaction between the organic content of the peat and the water added, this also reduced the pH and prevented the binder from activating. Although as is discussed in the next section this was only part of the reason for the non activation of the Red Gypsum-GGBS binder in the peat, the structure of the soil also having significant effect.

The peat samples offer the clearest demonstration of how the soil structure or soil skeleton is a strong factor in the effectiveness of the binder. The peat is composed almost exclusively of fine fibres meaning that even when compacted there are significant numbers of voids with the soil, especially when the soil is ex-situ with no confining pressure on the soil. In soils of this type the amount of contact between the fibres is significantly lower than in a soil composed of more spherical particles. Reduced amount of surface contact provides less area for the binder to bind particles together. Curing the samples under a confining pressure was an attempt to overcome this however this proved time consuming, inefficient and ultimately ineffective. Soil structure also had an influence on the higher strength achieved by the silty sand samples when compared to the Glacial Till and London Clay samples. The Silty Sand is a much courser soil than either the Glacial Till or the London Clay, this allows the soil to behave more like a mortar as the binder cements the course sand grains

together with few fines in the matrix. In the Glacial Till and London Clay the dominant particle size is fine and therefore the binder is essentially cementing clay and silt particles together which have a much lower strength than the sand.

In light of the results of this study a number of recommendations should be made. Since it has been clearly identified that the pH of the soil is critical in the development of strength and that the test described in BS 1377 (BSI, 1990) is not a suitable method of assessing this when Red Gypsum – GGBS binders are to be used the long term effect of the agent used to increase the pH should be investigated and the quantity of that agent should be sufficient to maintain the pH above 10.5 for at least 56 days.

In conclusion, this testing programme has been successful in proving the potential of using Red Gypsum – GGBS binders to improve a range of soils, there are however a range of issues that need to be addressed before the binder can be used commercially.

The rate of hardening is slow when compared to Portland cement, since Portland cement contains lime it would be desirable to use Portland cement in the place of lime alone to increase the pH, this would then also provide a fast initial strength gain, albeit lower than using Portland cement alone, to the improved soil.

Using this technique it would be desirable to repeat the testing programme on the London Clay and the un-soaked Glacial Till. In this programme, due to time and cost demands it was not possible to conduct durability testing of the improved soil samples. If Red Gypsum – GGBS binders are to be used commercially then the durability of the improved soils must be proved. A test programme involving freeze thaw and wet dry durability tests should therefore be conducted.

Whilst the mineralogical testing was successful in indicating the presence of minerals such as ettringite it was unsuccessful in proving the presence of C-S-H. A further testing programme should be conducted making use of other mineralogical analysis techniques to identify what minerals are being formed with the samples. Conducting SEM analysis on broken sections of the improved soil rather than polished sections may aid this, such as the investigation performed successfully by Al-Tabbaa and Evans (1999).

# Chapter 6

# Blockwork Results

# 6. Blockwork Results

## 6.1 Introduction

The aim of this programme was to test a variety of Red Gypsum / GGBS concrete mixes and compare their strength and strength development with Portland cement mixes in order to see if there is the potential for Red Gypsum binders to be used in the production of paving blocks.

The research programme was split into 2 parts. After a total of 15 suitable mixes (including controls) were selected, an initial programme of workability testing was conducted in order to select suitable water contents for the next phase of research. Because any mixes developed would need to be suitable for existing block making plant and equipment it was decided that mixes should be chosen on the basis of workability rather than water content.

In the second phase of the research cubes were made from the mixes at 3 different water contents (chosen on the basis of results from the first workability phase) and tested for compressive strength at 1, 7, and 28 days. Non destructive testing (Ultrasonic Pulse Velocity (UPv)) was conducted on the cubes at 7 and 28 days, and bulk densities were also checked at 1, 7, and 28 days

In light of further binder development testing conducted at Huntsman Tioxide Ltd (see data contained in Appendix D) and the necessity of rapid hardening to facilitate easier handling, two new gypsum binders were selected for this purpose, each containing a proportion of Portland Cement to ensure swift hardening. These were then compared with a Portland cement binder.

The mixing, curing and testing procedures are detailed in Chapter 3.

### 6.1.1 Binder Types (percentages by dry weight)

Three binders were used in this testing programme these were,

Type A = 27% Red Gypsum, 67% Ground granulated blastfurnace slag, 6% Portland cement*
Type B = 57% Red Gypsum, 37% Ground granulated blastfurnace slag, 6% Portland cement*
Portland = 100% Portland cement

*Red Gypsum was added to the mix as filter cake (wet), the water content of the Red Gypsum being subtracted from the water added to the mix.

### 6.1.2 Mixes

The ratio of cement to sand to aggregate (where present) was 1:2:4 by dry weight.
In the mixes where ochre was used, 20% of the dry weight of the binder was added. Table 6.1 shows the ingredients of the mixes tested. For ease of reference in graphs the table also shows the reference of each sample when displayed in graph legends, the format used is mix number / binder type / sand type / aggregate (if present) / ochre (if present).

Table 6.1, Mixes tested, (*20% of dry weight of binder added), the second column shows the way the mixes are referred to in graph legends, see section 6.1.2 for details.

| Mix | Graph Legend Ref | Binder Type | Sand | Aggregate | Ochre |
|---|---|---|---|---|---|
| 1 | 1/A/Ic | A | ICON | None | None |
| 2 | 2/B/Ic | B | ICON | None | None |
| 3 | 3/A/Bu | A | Building | None | None |
| 4 | 4/B/Bu | B | Building | None | None |
| 5 | 5/A/Ic/Ag | A | ICON | 10 mm | None |
| 6 | 6/B/Ic/Ag | B | ICON | 10 mm | None |
| 7 | 7/A/Bu/Ag | A | Building | 10 mm | None |
| 8 | 8/B/Bu/Ag | B | Building | 10 mm | None |
| 9 | 9/A/Ic/Ag/och | A | ICON | 10 mm | Yes* |
| 10 | 10/B/Bu/Ag/och | B | ICON | 10 mm | Yes* |
| Control 1 | C1/P/Ic | Portland | ICON | None | None |
| Control 2 | C2/P/Bu | Portland | Building | None | None |
| Control 3 | C3/P/Ic/Ag | Portland | ICON | 10 mm | None |
| Control 4 | C4/P/Bu/Ag | Portland | Building | 10 mm | None |
| Control 5 | C5/P/Ic/Ag/och | Portland | ICON | 10 mm | Yes* |

## 6.2 Workability Results

### 6.2.1 Mortar Mixes

Figure 6.1 shows slump workability plotted against water content of the mortar mixes.

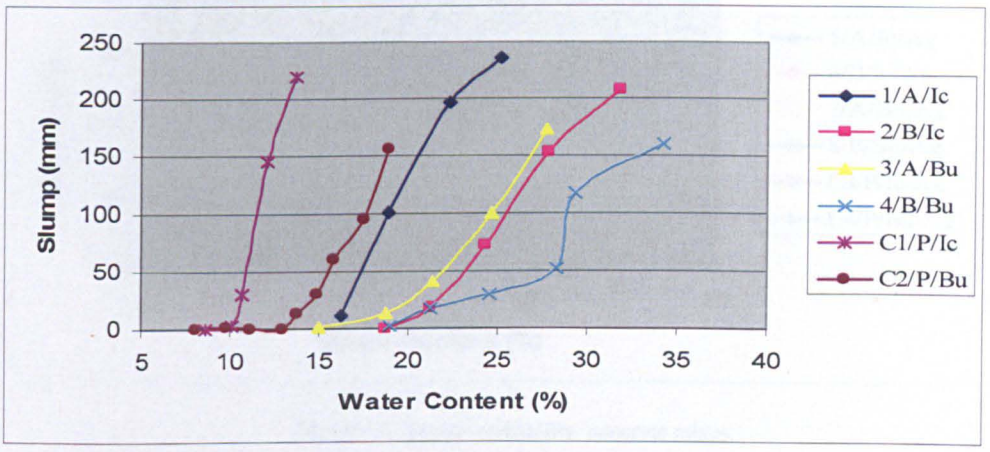



Figure 6.1, Slump workability, mortar mixes.

As can be seen in Figure 6.1, in the case of mortar samples, cubes made with Red Gypsum binders required more water to attain the same workability as Portland cement samples. Cubes made with Binder A required less water than Binder B.

Samples containing ICON sand in the place of building sand required less water to attain the same workability.

### 6.2.2 Concrete Mixes

Figure 6.2 shows slump workability of the concrete mixes plotted against water content.

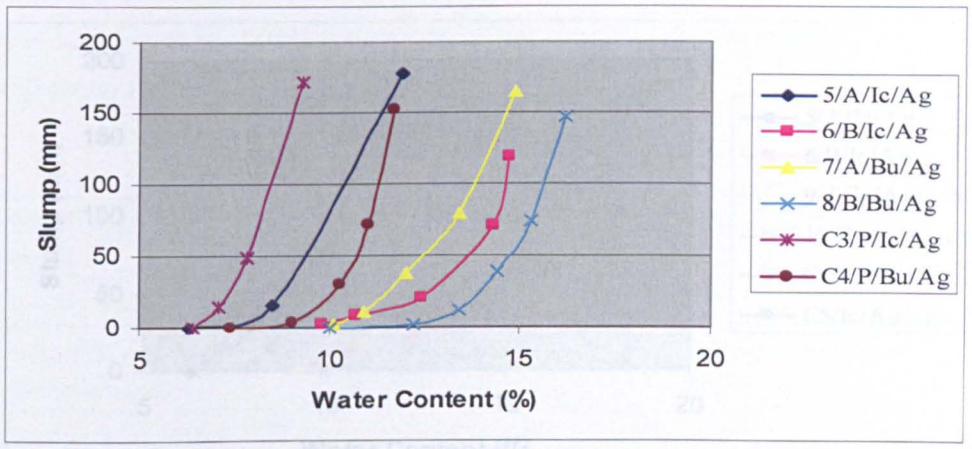



Figure 6.2, Slump workability, concrete mixes.

Figure 6.2 indicates that, as with the mortar samples, concrete samples containing ICON sand in the place of building sand required less water to attain the same workability.

Taking the effect of the ICON sand into account, samples prepared with Portland cement binder required less water to become workable than samples with the gypsum binders. Of the gypsum binders Binder A required less water than Binder B.

### 6.2.3 Ochre

Figure 6.3 shows slump workability of the ochre mixes plotted against water content.

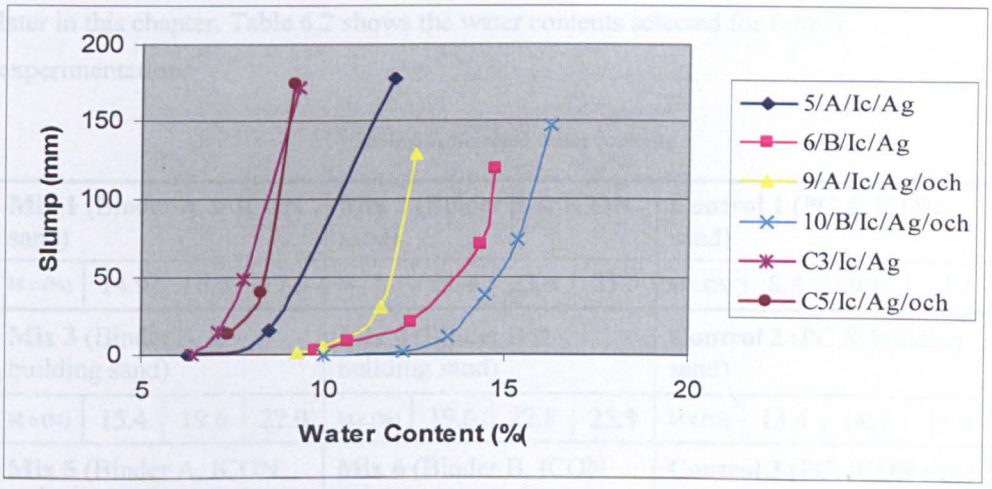



Figure 6.3, Slump Workability, comparison of samples with and without ochre.

Figure 6.3 indicates the addition of ochre to concrete mixes made little difference to the workability in the case of mixes made with a Portland cement binder.

In the mixes made with Red Gypsum binder the addition of ochre caused a loss of workability.

Taking the effect of the ochre into account mixes prepared with Binder A required less water than Binder B.

### 6.2.4 Selected Water Contents for Cube Mixes

On the basis of the workability results the following water contents were selected for the cube mixing. The justification for the choice of water contents is explained in Section 6.7 later in this chapter. Table 6.2 shows the water contents selected for further experimentation.

Table 6.2, Selected water contents

| **Mix 1** (Binder A & ICON sand) | | | | **Mix 2** (Binder B & ICON sand) | | | | **Control 1** (PC & ICON sand) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 14.9 | 16.6 | 17.5 | M.c.(%) | 21.4 | 23.9 | 25.5 | M.c.(%) | 8.8 | 9.6 | 11.1 |
| **Mix 3** (Binder A & building sand) | | | | **Mix 4** (Binder B & building sand) | | | | **Control 2** (PC & building sand) | | | |
| M.c.(%) | 15.4 | 19.6 | 22.0 | M.c.(%) | 19.6 | 22.8 | 25.9 | M.c.(%) | 13.4 | 14.7 | 15.6 |
| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand & pea gravel) | | | | **Control 3** (PC, ICON sand and pea gravel) | | | |
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| **Mix 7** (Binder A, building sand & pea gravel) | | | | **Mix 8** (Binder B, building sand & pea gravel) | | | | **Control 4** (PC building sand & pea gravel) | | | |
| M.c.(%) | 11.1 | 12.7 | 13.5 | M.c.(%) | 11.6 | 12.6 | 13.7 | M.c.(%) | 7.5 | 8.8 | 10.4 |
| **Mix 9** (Binder A, ICON sand, pea gravel and ochre) | | | | **Mix 10** (Binder B, ICON sand, pea gravel and ochre) | | | | **Control 5** (PC, ICON sand, pea gravel and ochre) | | | |
| M.c.(%) | 10.7 | 11.9 | 12.4 | M.c.(%) | 12.5 | 15.4 | 16.4 | M.c.(%) | 7.2 | 7.7 | 9.2 |

### 6.3 Uniaxial Compressive Strength

#### 6.3.1 Mortar Cubes

**Cubes Made with ICON Sand**

Figure 6.4 shows the UCS the mortar cubes made with ICON sand against curing time.

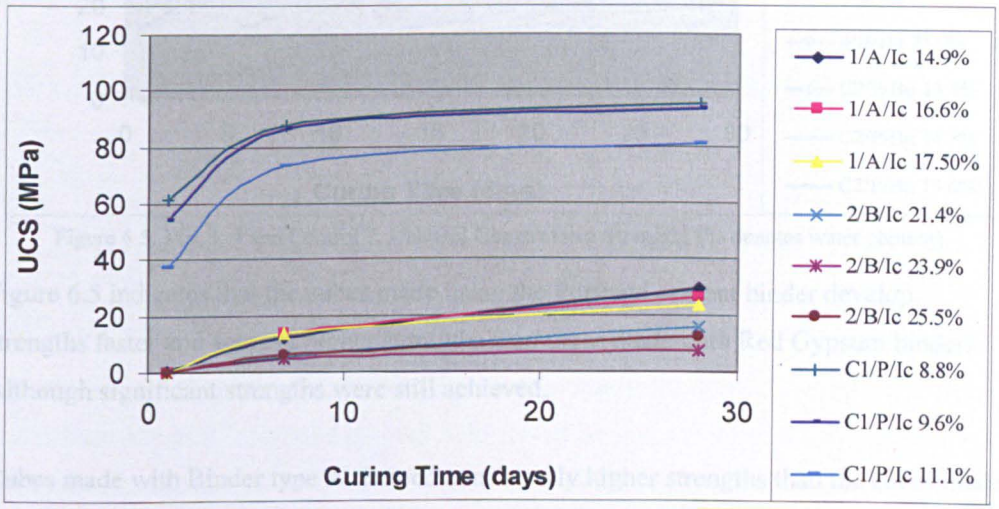



Figure 6.4, Mix 1, 2 and Control 1, Uniaxial Compressive Strengths (% denotes water content)

Figure 6.4 indicates that the cubes made using the Portland cement binder develop strengths faster and achieve higher strengths than those made with Red Gypsum binders. Although significant strengths were still achieved using the Red Gypsum binders. Cubes made with Binder Type A showed consistently higher strengths than the cubes made with Binder B. In general cubes made with lower water contents showed higher shear strengths.

**Cubes made with Building sand**

Figure 6.5 shows the UCS of the mortar cubes made with building sand against curing times.

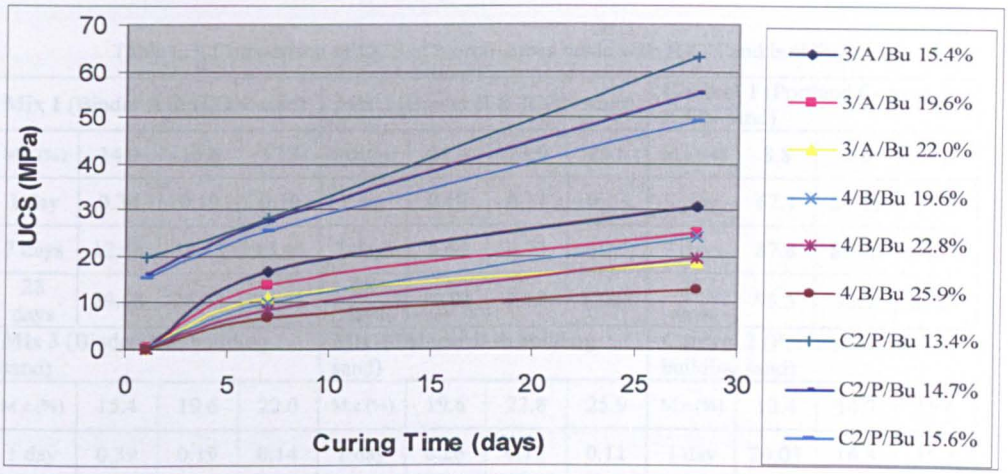



Figure 6.5, Mix 3, 4 and Control 2, Uniaxial Compressive Strengths (% denotes water content).

Figure 6.5 indicates that the cubes made using the Portland cement binder develop strengths faster and achieve higher strengths than those made with Red Gypsum binders. Although significant strengths were still achieved.

Cubes made with Binder type A showed consistently higher strengths than the cubes made with Binder B. Cubes made with lower water contents showed higher shear strengths.

**Comparison Between ICON Sand and Building Sand**

Figure 6.6 shows a comparison of the samples made with the different sands in the study.

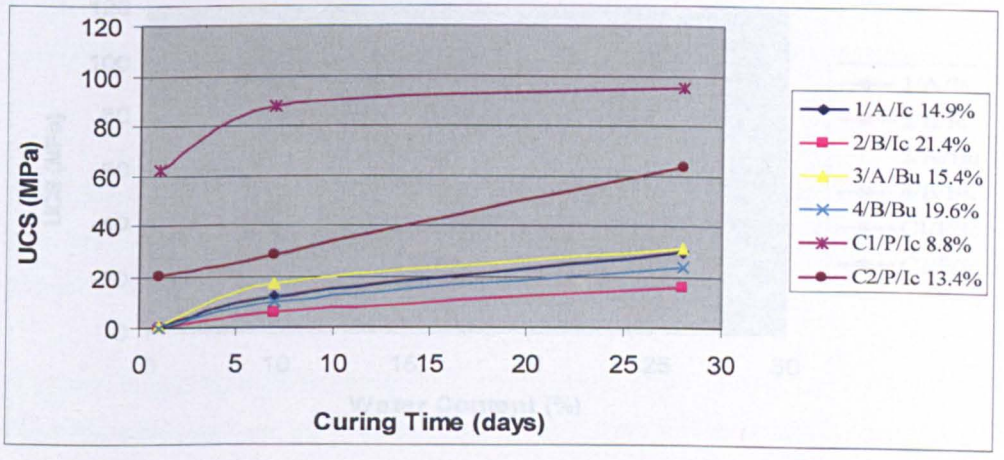



Figure 6.6, Comparison of UCS of mortar cubes made with ICON and building sand (% denotes water content).

Table 6.3, Comparison of UCS of mortar cubes made with ICON and building sand.

| **Mix 1** (Binder A & ICON sand) | | | | **Mix 2** (Binder B & ICON sand) | | | | **Control 1** (Portland Cement & ICON sand) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 14.9 | 16.6 | 17.5 | M.c.(%) | 21.4 | 23.9 | 25.5 | M.c.(%) | 8.8 | 9.6 | 11.1 |
| 1 day | 0.34 | 0.19 | 0.19 | 1 day | 0.17 | 0.11 | 0.13 | 1 day | 62.1 | 54.25 | 38.8 |
| 7 days | 12.34 | 13.43 | 13.40 | 7 days | 5.64 | 4.73 | 6.15 | 7 days | 87.8 | 86.82 | 73.7 |
| 28 days | 29.68 | 26.44 | 23.45 | 28 days | 16.01 | 7.12 | 12.65 | 28 days | 95.5 | 93.5 | 81.27 |
| **Mix 3** (Binder A & building sand) | | | | **Mix 4** (Binder B & building sand) | | | | **Control 2** (Portland Cement & building sand) | | | |
| M.c.(%) | 15.4 | 19.6 | 22.0 | M.c.(%) | 19.6 | 22.8 | 25.9 | M.c.(%) | 13.4 | 14.7 | 15.6 |
| 1 day | 0.39 | 0.19 | 0.14 | 1 day | 0.26 | 0.15 | 0.11 | 1 day | 20.03 | 16.3 | 15.5 |
| 7 days | 17.02 | 13.71 | 11.18 | 7 days | 10.09 | 8.51 | 6.75 | 7 days | 28.56 | 27.6 | 25.8 |
| 28 days | 30.86 | 24.86 | 18.45 | 28 days | 24.41 | 19.61 | 12.82 | 28 days | 63.36 | 55.4 | 49.3 |

As can be seen in Figure 6.6 and Table 6.3 cubes prepared using Binder A (mixes 1 and 3) and Binder B (mixes 2 and 4) exhibited little change in strength when ICON sand was used in place of building sand. In the case of cubes prepared using Portland cement as a binder however cubes containing ICON sand were significantly stronger than those containing building sand, although it should be noted that there is also a significant difference in water contents which could be responsible for the difference.

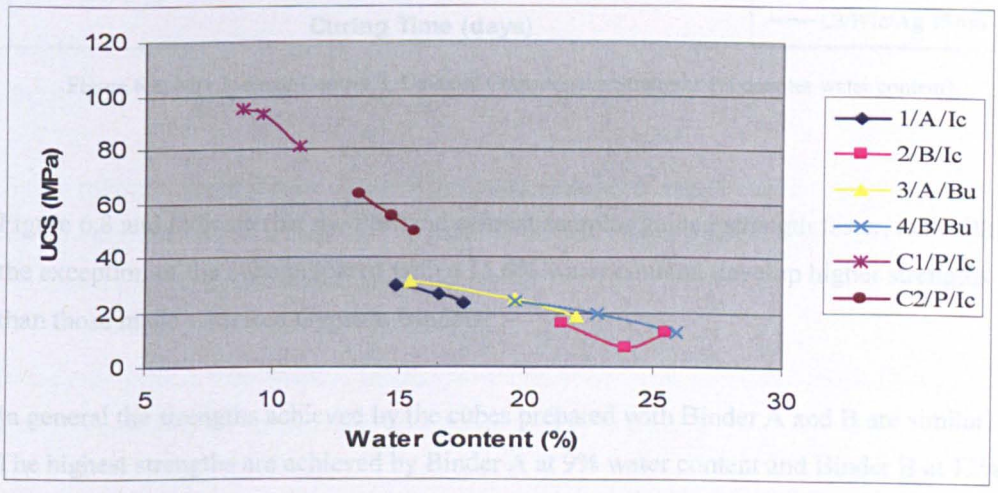



Figure 6.7, Effect of Water content on UCS of 28 day mortar samples.

By plotting UCS against water content for all mixes, the relationship between strength and water content can be investigated. Figure 6.7 clearly illustrates this relationship. It shows that water content has had a significant effect on the strength of the samples, in that water content is inversely proportional to water content.

### 6.3.2 Concrete Cubes

**Concrete cubes made with ICON sand**

Figure 6.8 shows the UCS of the samples with ICON sand against curing time.

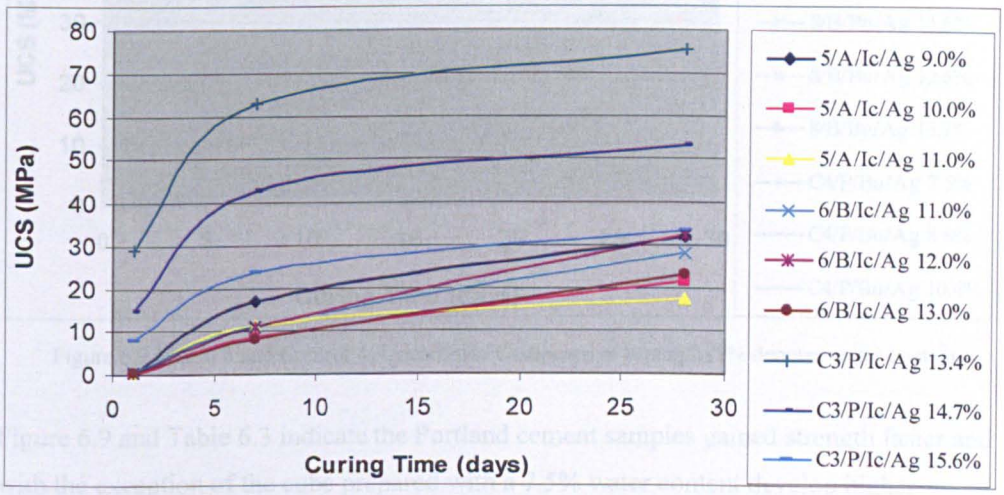



Figure 6.8, Mix 5, 6 and Control 3, Uniaxial Compressive Strengths (% denotes water content).

Figure 6.8 and indicate that the Portland cement samples gained strength faster, and with the exception of the cube prepared with a 15.6% water content, develop higher strengths than those made with Red Gypsum Binders.

In general the strengths achieved by the cubes prepared with Binder A and B are similar. The highest strengths are achieved by Binder A at 9% water content and Binder B at 12% water content.

In general lower water contents generated higher strengths in cubes made with all binders.

**Concrete Cubes made with Building Sand**

Figure 6.9 shows the strengths achieved by the cubes made with building sand against curing time.

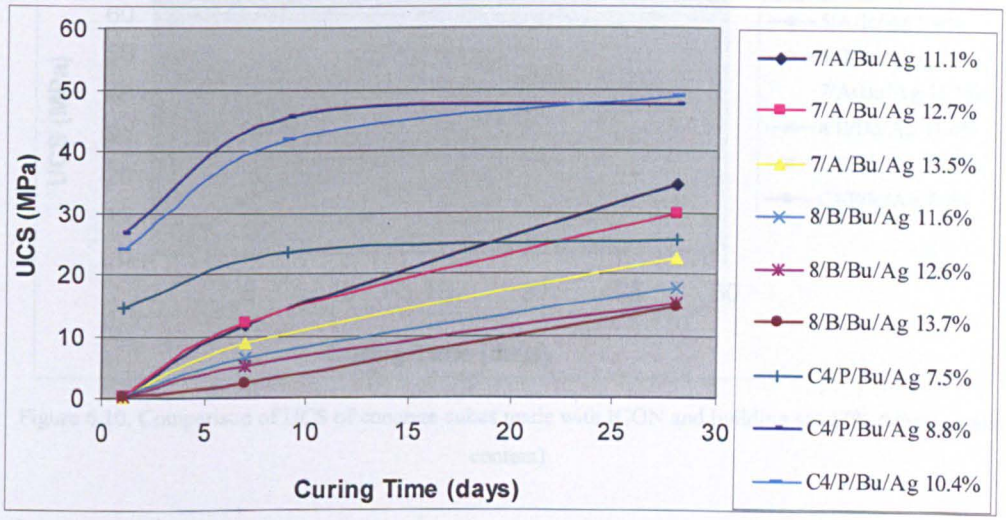



Figure 6.9 Mix 7, 8 and Control 4, Unconfined Compressive Strengths (% denotes water content).

Figure 6.9 and Table 6.3 indicate the Portland cement samples gained strength faster and with the exception of the cube prepared with a 7.5% water content develop higher strengths than those made with Red Gypsum Binders.

Cubes prepared using Binder A developed strengths faster and achieved higher strengths than those achieved by Binder B. The highest strength of a Binder A cube being nearly twice as strong as the strongest Binder B cube.

In the cubes made with gypsum binders lower water contents generated higher strengths; the reverse is true for cubes made with Portland cement binders.

**Comparison Between ICON Sand and Building Sand**

Figure 6.10 and Table 6.4 comparison of strengths achieved by the different concrete mixes against curing time.

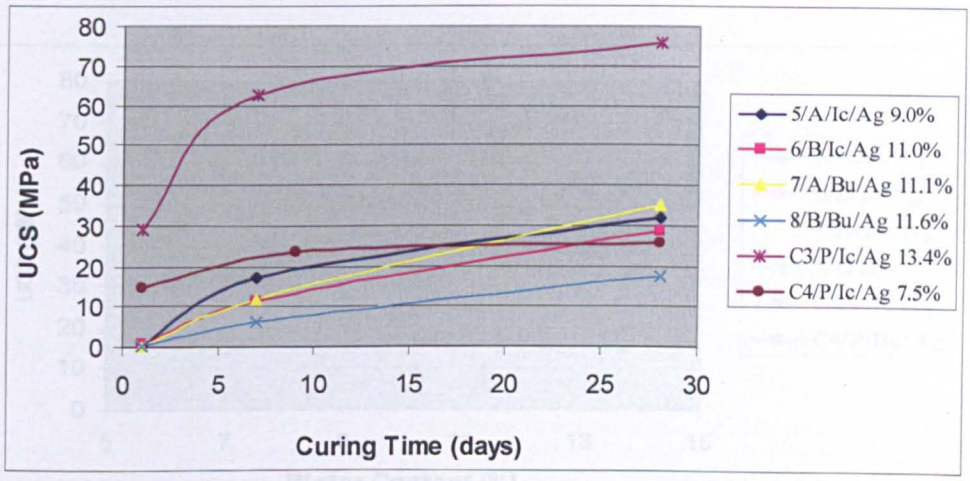



Figure 6.10, Comparison of UCS of concrete cubes made with ICON and building sand (% denotes water content).

Table 6.4, Comparison of UCS of concrete cubes made with ICON and building sand (values in MPa).

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand & pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| 1 day | 0.56 | 0.17 | 0.25 | 1 day | 0.70 | 0.5 | 0.3 | 1 day | 28.1 | 14.8 | 8.02 |
| 7 days | 17.15 | 8.59 | 11.32 | 7 days | 11.50 | 11.0 | 8.0 | 7 days | 62.9 | 42.9 | 23.97 |
| 28 days | 31.84 | 21.29 | 17.36 | 28 days | 28.50 | 32.2 | 23.0 | 28 days | 75.6 | 53.3 | 33.5 |
| **Mix 7** (Binder A, building sand & pea gravel) | | | | **Mix 8** (Binder B, building sand & pea gravel) | | | | **Control 4** (Portland Cement, building sand & pea gravel) | | | |
| M.c.(%) | 11.1 | 12.7 | 13.5 | M.c.(%) | 11.6 | 12.6 | 13.7 | M.c.(%) | 7.5 | 8.8 | 10.4 |
| 1 day | 0.2 | 0.17 | 0.09 | 1 day | 0.32 | 0.19 | 0.15 | 1 day | 14.62 | 26.8 | 24.2 |
| 7 days | 11.91 | 12.23 | 8.87 | 7 days | 6.38 | 5.05 | 2.27 | 7 days | 23.7 | 45.7 | 41.98 |
| 28 days | 34.71 | 29.97 | 22.46 | 28 days | 17.65 | 15.14 | 14.59 | 28 days | 25.5 | 47.6 | 48.97 |

As can be seen in Figure 6.10 and Table 6.4 cubes prepared using Binder A (mixes 5 and 7) exhibited little change in strength when ICON sand was used in place of building sand.

In the case of cubes prepared using Binder B (mixes 6 and 8) cubes made with ICON were slightly stronger despite being prepared at similar water contents. In the case of cubes made with Portland cement the cubes containing ICON sand were twice as strong as the ones containing building sand, although there was a large difference in water content also.

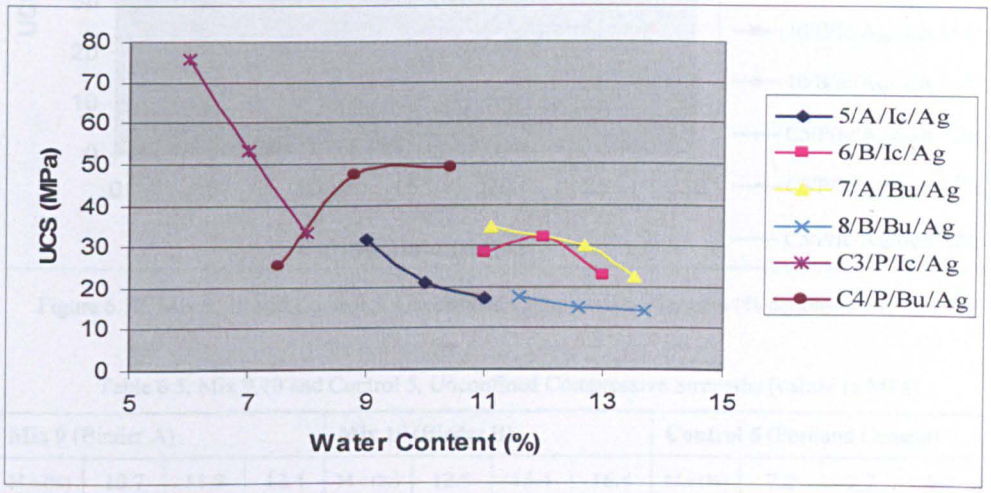



Figure 6.11, Affect of Water content on UCS of 28 day concrete samples.

As can be seen in Figure 6.11, with the exception of mix 6 and control 4, samples became stronger with lower water contents. There is a much more pronounced difference in strength between the different concrete mixes than the mortar mixes.

### 6.3.3 Concrete Cubes made with Ochre Additions

Figure 6.12 and Table 6.5 shows a comparison of the strengths of cubes made with and without ochre additions against curing time.

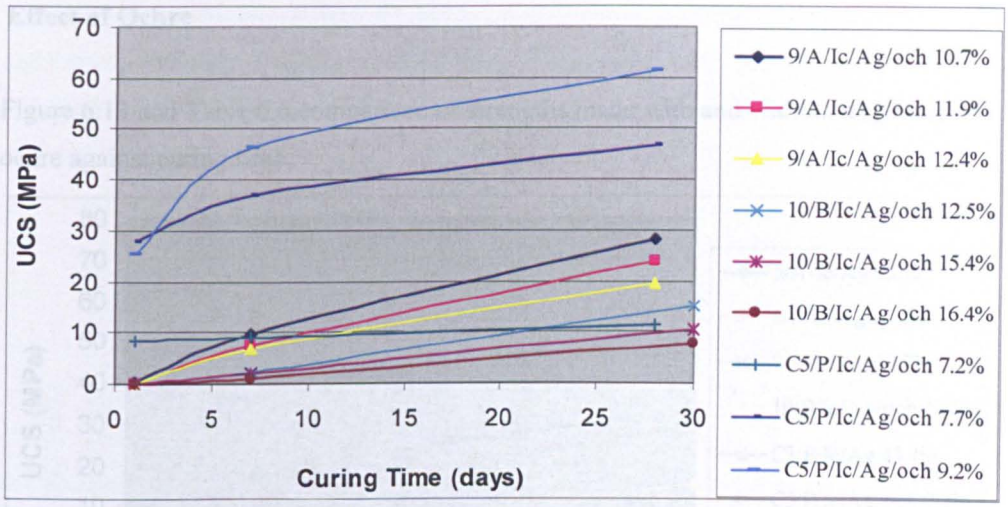



Figure 6.12, Mix 9, 10 and Control 5, Unconfined Compressive Strengths (% denotes water content).

Table 6.5, Mix 9,10 and Control 5, Unconfined Compressive Strengths (values in MPa).

| **Mix 9** (Binder A) | | | | **Mix 10** (Binder B) | | | | **Control 5** (Portland Cement) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 10.7 | 11.9 | 12.4 | M.c.(%) | 12.5 | 15.4 | 16.4 | M.c.(%) | 7.2 | 7.7 | 9.2 |
| 1 day | 0.28 | 0.15 | 0.13 | 1 day | 0.16 | 0.12 | 0.06 | 1 day | 8.5 | 27.8 | 25.4 |
| 7 days | 9.62 | 7.55 | 6.83 | 7 days | 2.3 | 1.96 | 0.99 | 7 days | 9.21 | 37.2 | 46.6 |
| 28 days | 28.11 | 24.02 | 19.59 | 28 days | 15.1 | 10.56 | 7.62 | 28 days | 11.43 | 46.8 | 61.7 |

Figure 6.12 and Table 6.5 show the OPC samples gained strength faster and with the exception of the cube prepared with a 7.2% water content develop higher strengths than those made with Red Gypsum Binders.

Cubes prepared using Binder A developed strengths faster and achieved higher strengths than those achieved by Binder B. The highest strength of a Binder A cube being nearly twice as strong as the strongest Binder B cube. In the cubes made with gypsum binders lower water contents generated higher strengths, the reverse is true for cubes made with PC binders. This is due to the PC samples being too dry for effective compaction to occur.

**Effect of Ochre**

Figure 6.13 and Table 6.6 comparison of strengths made with and without additions of ochre against curing time.

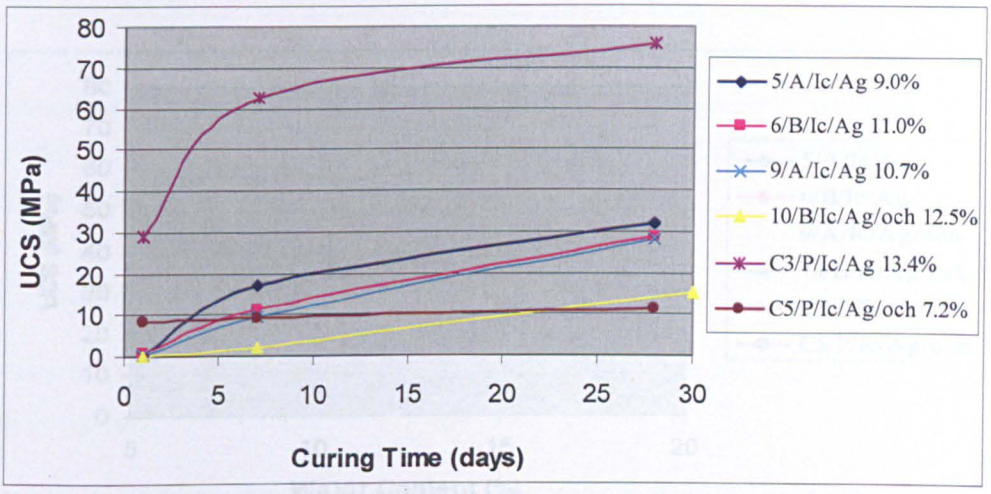



Figure 6.13, Comparison of UCS of concrete cubes made with and without ochre (% denotes water content).

Table 6.6, Comparison of UCS of concrete cubes made with and without ochre (values in MPa).

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand & pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| 1 day | 0.56 | 0.17 | 0.25 | 1 day | 0.70 | 0.5 | 0.3 | 1 day | 28.1 | 14.8 | 8.02 |
| 7 days | 17.15 | 8.59 | 11.32 | 7 days | 11.50 | 11.0 | 8.0 | 7 days | 62.9 | 42.9 | 23.97 |
| 28 days | 31.84 | 21.29 | 17.36 | 28 days | 28.50 | 32.2 | 23.0 | 28 days | 75.6 | 53.3 | 33.5 |
| **Mix 9** (Binder A, ICON sand, pea gravel and ochre) | | | | **Mix 10** (Binder B, ICON sand, pea gravel and ochre) | | | | **Control 5** (Portland Cement, ICON sand, pea gravel and ochre) | | | |
| M.c.(%) | 10.7 | 11.9 | 12.4 | M.c.(%) | 12.5 | 15.4 | 16.4 | M.c.(%) | 7.2 | 7.7 | 9.2 |
| 1 day | 0.28 | 0.15 | 0.13 | 1 day | 0.16 | 0.12 | 0.06 | 1 day | 8.5 | 27.8 | 25.4 |
| 7 days | 9.62 | 7.55 | 6.83 | 7 days | 2.3 | 1.96 | 0.99 | 7 days | 9.21 | 37.2 | 46.6 |
| 28 days | 28.11 | 24.02 | 19.59 | 28 days | 15.1 | 10.56 | 7.62 | 28 days | 11.43 | 46.8 | 61.7 |

The cubes prepared with Binder A (mixes 5 and 9) show little change when ochre is added , however in the case of cubes prepared using Binder B (mixes 6 and 10) there is a significant drop in strength when ochre is added. This can also be seen in the Portland cement control samples.

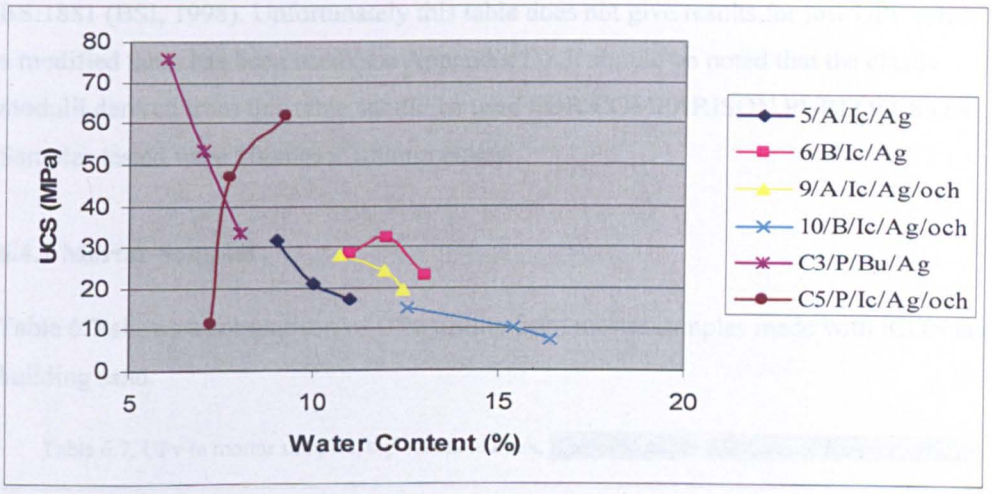



Figure 6.14, Affect of Water content on UCS of 28 day concrete/ochre samples.

Despite mix 9 having a higher water content than mix 5 they both achieved similar strengths (see Figure 6.14). Figure 6.14 also illustrates that the Portland cement samples without ochre reduced in strength with increasing water content, whereas Portland cement samples with ochre gained strength with increasing water content.

### 6.4 Ultrasonic Pulse Velocity (UPv)

The speed at which ultrasonic pulses travel through concrete give an indication of the stiffness of the concrete. The speed of the waves in measured in kilometres per second, which can be converted to dynamic modulus of elasticity using a table contained in BS:1881 (BSI, 1998). Unfortunately this table does not give results for low UPv values, so a modified table has been used (see Appendix D). It should be noted that the elastic modulii derived from this table should be used FOR COMPARISON PURPOSES ONLY. Samples tested were 100mm x 100mm cubes.

#### 6.4.1 Mortar Samples

Table 6.7 shows a comparison of UPv results from mortar samples made with ICON and building sand.

Table 6.7, UPv in mortar samples, top values in km/s, lower values are Modulus of Elasticity (GPa).

| **Mix 1** (Binder A & ICON sand) | | | | **Mix 2** (Binder B & ICON sand) | | | | **Control 1** (Portland Cement & ICON sand) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 14.9 | 16.6 | 17.5 | M.c.(%) | 21.4 | 23.9 | 25.5 | M.c.(%) | 8.8 | 9.6 | 11.1 |
| 7 days | 2.91 | 3.07 | 3.00 | 7 days | 2.62 | 2.59 | 2.56 | 7 days | 4.42 | 4.40 | 4.27 |
| | 18.5 | 19.5 | 19 | | 17 | 17 | 17 | | 36 | 36 | 34 |
| 28 days | 3.50 | 3.48 | 3.37 | 28 days | 3.05 | 3.01 | 3.06 | 28 days | 4.58 | 4.48 | 4.37 |
| | 22.5 | 22.5 | 22 | | 19 | 19 | 19 | | 42 | 39 | 36 |
| **Mix 3** (Binder A & building sand) | | | | **Mix 4** (Binder B & building sand) | | | | **Control 2** (Portland Cement & building sand) | | | |
| M.c.(%) | 15.4 | 19.6 | 22.0 | M.c.(%) | 19.6 | 22.8 | 25.9 | M.c.(%) | 13.4 | 14.7 | 15.6 |
| 7 days | 2.69 | 3.62 | 2.96 | 7 days | 2.83 | 2.74 | 2.58 | 7 days | 3.86 | 3.74 | 3.60 |
| | 17.5 | 24 | 19 | | 18 | 18 | 17 | | 27.5 | 25 | 24 |
| 28 days | 3.47 | 3.29 | 3.34 | 28 days | 2.80 | 2.72 | 2.25 | 28 days | 3.96 | 2.58 | 3.48 |
| | 22.5 | 21 | 21 | | 18 | 17.5 | - | | 29 | 17 | 24 |

The UPv results from the mortar cubes (see Table 6.7) show that as with Unconfined Compressive Strengths the Portland cement cubes gained stiffness faster and attained higher stiffness (approx 25% stiffer). However, Red Gypsum samples also achieved

significant stiffness, Binder A samples attaining higher stiffness than Binder B, again mirroring compressive strength results.

In the Portland cement samples replacing standard building sand with ICON raised the measured stiffness by approximately 15% in the stiffest (lowest water content) samples.

In the case of samples made with Red Gypsum binder there was little change in stiffness with the change in sand in the cubes made with Binder A. In the Binder B cubes the replacement of building sand with ICON sand increased the stiffness by approximately 10%.

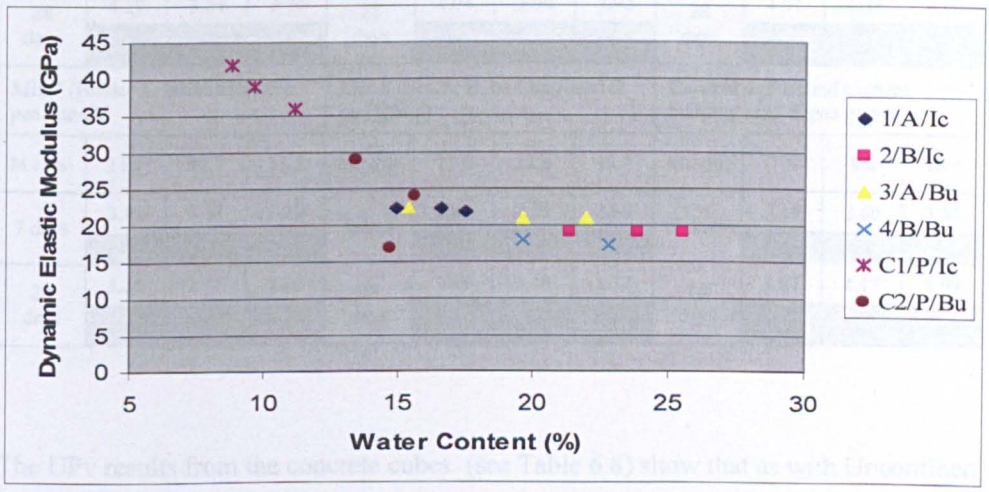



Figure 6.15, Mortar mixes dynamic elastic modulus.

Figure 6.15 illustrates that in general in the mortar mixes are stiffer when mixed a lower water contents.

### 6.4.2 Concrete Samples

Table 6.8 shows a comparison of UPv results from concrete samples.

Table 6.8, UPv in concrete samples, values in km/s. , lower values are Modulus of Elasticity (GPa).

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand & pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| 7 days | 3.80 | 3.15 | 3.53 | 7 days | 3.30 | 2.59 | 3.61 | 7 days | 4.05 | 4.05 | 3.87 |
| | 26 | 20 | 23 | | 21.2 | 17 | 24 | | 29 | 29 | 27.5 |
| 28 days | 4.29 | 3.84 | 3.70 | 28 days | 4.04 | 3.90 | 3.92 | 28 days | 4.57 | 4.39 | 4.06 |
| | 34 | 26 | 25 | | 29 | 27.5 | 27.5 | | 42 | 36 | 29 |
| **Mix 7** (Binder A, building sand & pea gravel) | | | | **Mix 8** (Binder B, building sand & pea gravel) | | | | **Control 4** (Portland Cement, building sand & pea gravel) | | | |
| M.c.(%) | 11.1 | 12.7 | 13.5 | M.c.(%) | 11.6 | 12.6 | 13.7 | M.c.(%) | 7.5 | 8.8 | 10.4 |
| 7 days | 3.41 | 3.33 | 3.20 | 7 days | 2.81 | 2.89 | 2.66 | 7 days | 3.45 | 3.60 | 3.55 |
| | 22 | 21.2 | 20.5 | | 18 | 18.5 | 17 | | 22.5 | 24 | 24 |
| 28 days | 3.35 | 2.57 | 2.20 | 28 days | 3.49 | 3.48 | 3.32 | 28 days | 3.87 | 4.17 | 3.99 |
| | 21.2 | 17 | - | | 23 | 25 | 21.2 | | 27.5 | 32 | 29 |

The UPv results from the concrete cubes (see Table 6.8) show that as with Unconfined Compressive Strengths the Portland cement cubes gained stiffness faster and attained higher ultimate stiffness (approx 10% stiffer). However, Red Gypsum samples also achieved significant stiffness, Binder A samples attaining higher stiffness than Binder B, mirroring compressive strength results.

In the Portland cement samples, replacing standard building sand with ICON raised the measured stiffness by approximately 18% in the stiffest (lowest water content) samples.

In the Red Gypsum samples there were also increases in stiffness when building sand was replaced by ICON sand, 28% in the case of Binder A, 16% in the case of Binder B in the stiffest (lowest water content samples).

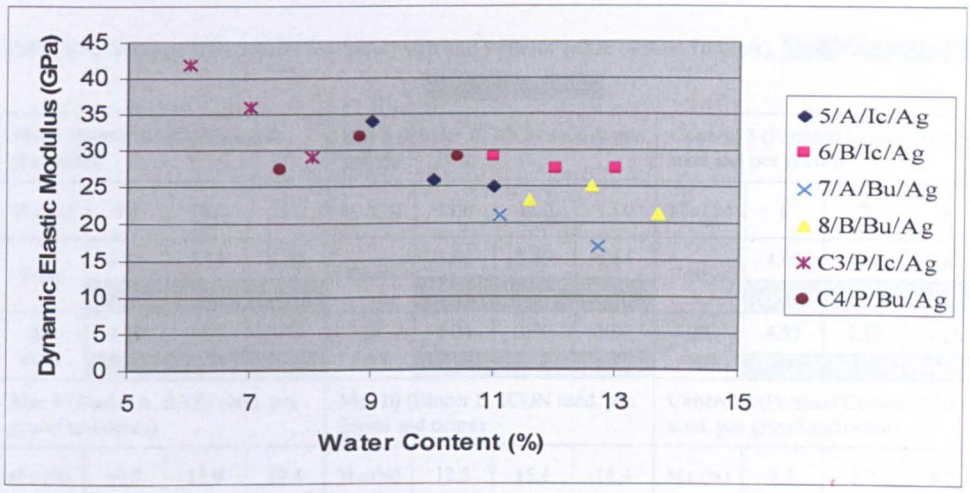



Figure 6.16, Concrete mixes dynamic elastic modulus.

Figure 6.16 illustrates that in general as with the mortar mixes, the concrete mixes are stiffer when mixed a lower water contents.

### 6.4.3 Ochre

Figure 6.17 and Table 6.9 shows a comparison of UPv results for mixes with and without ochre additions.

Table 6.9, Average UPv results for cubes with and without ochre (values in km/s), lower values are Modulus of Elasticity (GPa).

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand & pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| 7 day | 3.80 | 3.15 | 3.53 | 7 days | 3.30 | 2.59 | 3.61 | 7 days | 4.05 | 4.05 | 3.87 |
| | 26 | 20 | 23 | | 21.2 | 17 | 24 | | 29 | 29 | 27.5 |
| 28 days | 4.29 | 3.84 | 3.70 | 28 days | 4.04 | 3.90 | 3.92 | 28 days | 4.57 | 4.39 | 4.06 |
| | 34 | 26 | 25 | | 29 | 27.5 | 27.5 | | 42 | 36 | 29 |
| **Mix 9** (Binder A, ICON sand, pea gravel and ochre) | | | | **Mix 10** (Binder B, ICON sand, pea gravel and ochre) | | | | **Control 5** (Portland Cement, ICON sand, pea gravel and ochre) | | | |
| M.c.(%) | 10.7 | 11.9 | 12.4 | M.c.(%) | 12.5 | 15.4 | 16.4 | M.c.(%) | 7.2 | 7.7 | 9.2 |
| 7 days | 3.29 | 3.17 | 3.11 | 7 days | 2.50 | 2.62 | 2.63 | 7 days | 3.21 | 3.97 | 3.82 |
| | 21.2 | 20.5 | 19.7 | | - | 17 | 17 | | 20.5 | 29 | 26 |
| 28 days | 3.88 | 3.82 | 3.77 | 28 days | 2.19 | 2.29 | 2.26 | 28 days | 2.62 | 4.17 | 3.95 |
| | 27.5 | 26 | 26 | | - | - | - | | 17 | 32 | 29 |

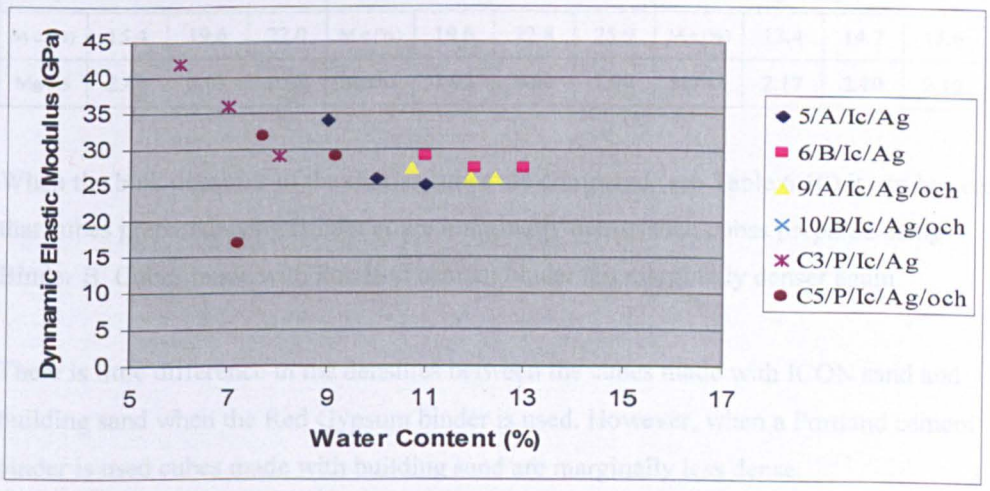



Figure 6.17, Ochre mixes dynamic elastic modulus.

Figure 6.17 illustrates that in general as with the mortar and concrete mixes, the ochre mixes are stiffer when mixed a lower water contents.

The addition of ochre to the mixes appears to have reduced the stiffness in all binder types, by 40% in Portland cement samples, 10% in Binder A samples, and 44% in Binder B samples in the stiffest (lowest water content samples).

### 6.5 Cube Density

#### 6.5.1 Mortar mixes

Table 6.10 shows the densities of the mortar mix cubes at 28 days curing.

Table 6.10, Bulk Densities of Mortar Cubes

| **Mix 1** (Binder A & ICON sand) | | | | **Mix 2** (Binder B & ICON sand) | | | | **Control 1** (Portland Cement & ICON sand) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 14.9 | 16.6 | 17.5 | M.c.(%) | 21.4 | 23.9 | 25.5 | M.c.(%) | 8.8 | 9.6 | 11.1 |
| Mg/m3 | 2.13 | 2.07 | 2.08 | Mg/m3 | 1.94 | 1.94 | 1.93 | Mg/m3 | 2.38 | 2.36 | 2.31 |

| **Mix 3** (Binder A & building sand) | | | | **Mix 4** (Binder B & building sand) | | | | **Control 2** (Portland Cement and building sand) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 15.4 | 19.6 | 22.0 | M.c.(%) | 19.6 | 22.8 | 25.9 | M.c.(%) | 13.4 | 14.7 | 15.6 |
| Mg/m3 | 2.00 | 2.03 | 2.08 | Mg/m3 | 1.97 | 1.94 | 1.90 | Mg/m3 | 2.17 | 2.10 | 2.10 |

When the bulk densities of the mortar cubes are compared (see Table 6.10) it can be seen that cubes prepared using Binder A are marginally denser than cubes prepared using Binder B. Cubes made with Portland cement binder are marginally denser again.

There is little difference in the densities between the cubes made with ICON sand and building sand when the Red Gypsum binder is used. However, when a Portland cement binder is used cubes made with building sand are marginally less dense.

### 6.5.2 Concrete Mixes

Table 6.11 shows the densities of the concrete cubes at 28 days curing.

Table 6.11, Bulk Densities of Concrete Cubes

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand and pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| Mg/m3 | 2.43 | 2.36 | 2.36 | Mg/m3 | 2.35 | 2.38 | 2.35 | Mg/m3 | 2.46 | 2.36 | 2.34 |

| **Mix 7** (Binder A, building sand and pea gravel) | | | | **Mix 8** (Binder B, building sand and pea gravel) | | | | **Control 4** (Portland Cement, building sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 11.1 | 12.7 | 13.5 | M.c.(%) | 11.6 | 12.6 | 13.7 | M.c.(%) | 7.5 | 8.8 | 10.4 |
| Mg/m3 | 2.26 | 2.24 | 2.24 | Mg/m3 | 2.20 | 2.21 | 2.21 | Mg/m3 | 2.18 | 2.32 | 2.31 |

In the case of the concrete cubes (see Table 6.11) again it can be seen that cubes prepared using Binder A are marginally denser than cubes prepared using Binder B. Cubes made with Portland cement binder are marginally denser again.

In the case of all three binder types cubes prepared with ICON sand are marginally more dense than those prepared with building sand.

### 6.5.3 Ochre

Table 6.12 shows the densities of concrete cubes with and without ochre additions.

Table 6.12, Bulk densities of concrete cubes with ochre

| **Mix 5** (Binder A, ICON sand & pea gravel) | | | | **Mix 6** (Binder B, ICON sand and pea gravel) | | | | **Control 3** (Portland Cement, ICON sand and pea gravel) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 9.0 | 10.0 | 11.0 | M.c.(%) | 11.0 | 12.0 | 13.0 | M.c.(%) | 6 | 7 | 8 |
| Mg/m3 | 2.43 | 2.36 | 2.36 | Mg/m3 | 2.35 | 2.38 | 2.35 | Mg/m3 | 2.46 | 2.36 | 2.34 |

| **Mix 9** (Binder A, ICON sand, pea gravel and ochre) | | | | **Mix 10** (Binder B ICON sand, pea gravel and ochre) | | | | **Control 5** (Portland Cement, ICON sand, pea gravel and ochre) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| M.c.(%) | 10.7 | 11.9 | 12.4 | M.c.(%) | 12.5 | 15.4 | 16.4 | M.c.(%) | 7.2 | 7.7 | 9.2 |
| Mg/m3 | 2.32 | 2.29 | 2.31 | Mg/m3 | 2.25 | 2.26 | 2.23 | Mg/m3 | 2.10 | 2.33 | 2.35 |

As can be seen from Table 6.12 the addition of ochre has not had a significant impact upon the bulk densities of the concrete cube samples, although there is a slight reduction in density.

### 6.5.4 Relationship between density, strength and water content

Figures 6.18 and 6.19 show the relationship between water content and dry density of the Red Gypsum and Portland cement cubes respectively.

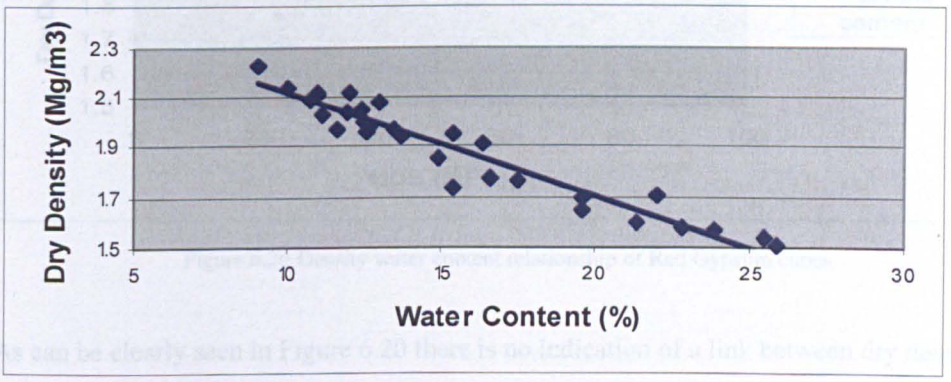



Figure 6.18 Density / water content relationship of Red Gypsum cubes.

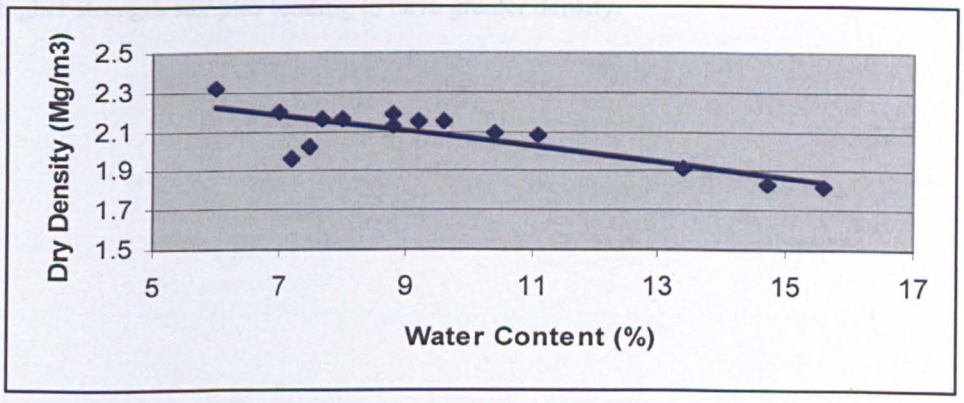



Figure 6.19 Density / water content relationship of Red Gypsum cubes.

Figure 6.18 and 6.19 clearly show that cubes with lower water contents have higher dry densities. Since dry density is often linked to strength Figure 6.20 shows the relationship between dry density and UCS.

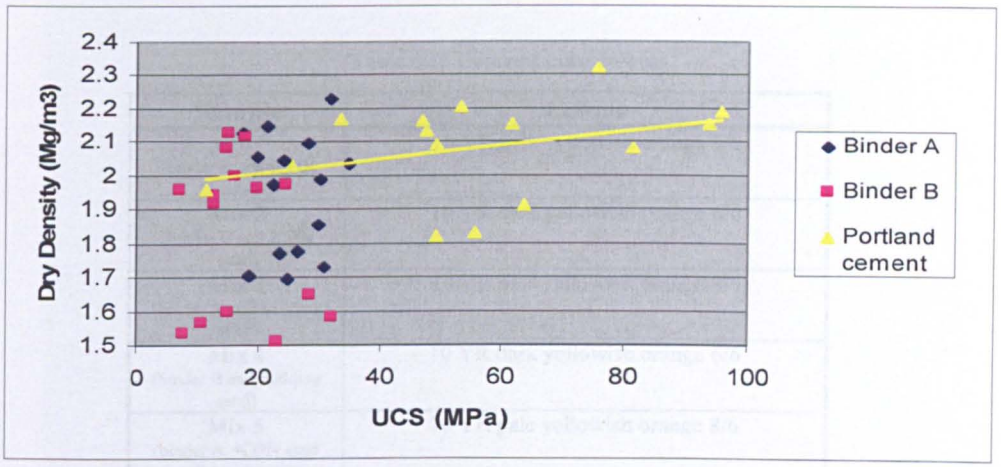



Figure 6.20 Density water content relationship of Red Gypsum cubes

As can be clearly seen in Figure 6.20 there is no indication of a link between dry density and the strength of the Red Gypsum cubes made with either binder A or binder B. There does appear to be a link between density and UCS in the Portland samples however, with higher strength samples tending to have greater density.

## 6.6 Colour

The colours of the blocks were compared and categorised using the Geological Society of America Rock Colour Chart (GSA, 1980), these are shown in Table 6.13.

Table 6.13 Concrete cube colours.

| Sample | Colour |
|---|---|
| Mix 1 (binder A and ICON sand) | 10 YR pale yellowish orange 8/6 |
| Mix 2 (binder B and ICON sand) | 10 YR dark yellowish orange 6/6 |
| Mix 3 (binder A and building sand) | 10 YR pale yellowish orange 8/6 |
| Mix 4 (binder B and building sand) | 10 YR dark yellowish orange 6/6 |
| Mix 5 (binder A, ICON sand and pea gravel) | 10 YR pale yellowish orange 8/6 |
| Mix 6 (binder B, ICON sand and pea gravel) | 10 YR dark yellowish orange 6/6 |
| Mix 7 (binder A, building sand and pea gravel) | 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 |
| Mix 8 (binder B, building sand and pea gravel) | 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 |
| Mix 9 (binder A, ICON sand and pea gravel) | 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 |
| Mix 10 (binder B, ICON sand, pea gravel and ochre) | 10 YR dark yellowish orange 6/6 |
| Control 1 (Portland cement and ICON sand) | N6 medium light grey |
| Control 2 (Portland cement and building sand) | 5GY light greenish grey 8/1 |
| Control 3 (Portland cement, ICON sand and pea gravel) | N6 medium light grey |
| Control 4 (Portland cement, building sand and pea gravel) | 5GY light greenish grey 8/1 |
| Control 5 (Portland cement, ICON sand, pea gravel and ochre) | 5YR moderate brown 4/4 |

### 6.6.1 Mortar Mixes

Figure 6.21 and Table 6.14 show the colours of the mortar cubes after curing.


Figure 6.21 Mortar cubes.

Table 6.14 Mortar cube colours.

| **Mix 1** (Binder A & ICON sand) | **Mix 2** (Binder B & ICON sand) | **Control 1** (Portland Cement & ICON sand) |
|---|---|---|
| 10 YR pale yellowish orange 8/6 | 10 YR dark yellowish orange 6/6 | N6 medium light grey |
| **Mix 3** (Binder A & building sand) | **Mix 4** (Binder B & building sand) | **Control 2** (Portland Cement and building sand) |
| 10 YR pale yellowish orange 8/6 | 10 YR dark yellowish orange 6/6 | 5GY light greenish grey 8/1 |

Mortar cubes made with Red Gypsum – GGBS binders were yellow orange in colour whereas the Portland cement cubes were grey (see Figure 6.21 and Table 6.14). Cubes made with Binder B were marginally darker than those made with Binder A. Changing the type of sand used from ICON to building sand did not have a significant effect on the colour of the Red Gypsum - GGBS cubes, However Portland cement cubes made with building sand were noticeably greener than those made with ICON sand.

### 6.6.2 Concrete Mixes

Figure 6.22 and Table 6.14 show the colours of the concrete cubes after curing.


Figure 6.22 Concrete Cube colours.

Table 6.14 Concrete Mixes.

| **Mix 5** (Binder A, ICON sand & pea gravel) | **Mix 6** (Binder B, ICON sand and pea gravel) | **Control 3** (Portland Cement, ICON sand and pea gravel) |
|---|---|---|
| 10 YR pale yellowish orange 8/6 | 10 YR dark yellowish orange 6/6 | N6 medium light grey |
| **Mix 7** (Binder A, building sand and pea gravel) | **Mix 8** (Binder B, building sand and pea gravel) | **Control 4** (Portland Cement, building sand and pea gravel) |
| 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 | 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 | 5GY light greenish grey 8/1 |

The concrete cubes made with Red Gypsum – GGBS binders were yellow orange and brown in colour, whereas the Portland cement cubes were grey (see Figure 6.22 and Table 6.14). Cubes made with Binder B were marginally darker than those made with Binder A. Cubes made with Red Gypsum – GGBS binder and building sand had some dark mottling although this could be an effect of oxidisation. Portland cement cubes made with building sand were noticeably greener than those made with ICON sand.

### 6.6.3 Ochre Mixes

Figure 6.23 and Table 6.15 show the colours of the concrete cubes with ochre additions.


Figure 6.23 Ochre Cube colours.

Table 6.15 Ochre cube colours.

| **Mix 5** (Binder A, ICON sand & pea gravel) | **Mix 6** (Binder B, ICON sand and pea gravel) | **Control 3** (Portland Cement, ICON sand and pea gravel) |
|---|---|---|
| 10 YR pale yellowish orange 8/6 | 10 YR dark yellowish orange 6/6 | N6 medium light grey |
| **Mix 9** (Binder A, ICON sand, pea gravel and ochre) | **Mix 10** (Binder B ICON sand, pea gravel and ochre) | **Control 5** (Portland Cement, ICON sand, pea gravel and ochre) |
| 10 YR pale yellowish orange 8/6 mottled with 5YR moderate brown 4/4 | 10 YR dark yellowish orange 6/6 | 5YR moderate brown 4/4 |

When ochre was added to the Red Gypsum – GGBS mixes there was not a significant effect on the colour; again the main factor affecting colour appeared to be the type of binder (and hence the percentage of Red Gypsum) in the mix. However, the ochre had a significant effect on the colour of the Portland cement cubes, changing them from grey to brown.

## 6.7 Discussion

### 6.7.1 Workability

More water is required to attain workability in gypsum mixes than mixes prepared with Portland cement. This applies to both mortar and concrete mixes (with either building or ICON sand, and with or without ochre). Binder A required less water to attain workability than Binder B. This indicates that gypsum is the controlling factor with regard to the water requirement to attain workable cement/concrete. Both Portland cement and GGBS are supplied in a powder form, the individual particles of each being rounded to sub-rounded in shape. By contrast, Red Gypsum particles are needle shaped and have a considerably higher surface area to volume ratio compared to the Portland cement and GGBS particles. Because of this more water becomes attached to Red Gypsum particles through surface tension than would attach to Portland cement or GGBS, and consequently requiring more water to make the mixes become a workable slurry.

Mixes containing ICON sand in place of building sand required less water to attain workability. This is also probably an effect of particle shape. The building sand grains are angular – sub angular, whereas the ICON sand is rounded – sub-rounded. There are also particles of $TiO_2$ contained within the ICON sand, which are angular; however, they do not appear to have had a significant effect on the workability due to making up less than 2% of the sand.

The addition of ochre to concrete mixes did not affect the workability significantly when the binder was Portland cement. However, in the case of cubes using Red Gypsum as the binding agent, the addition of ochre caused a loss of workability. The reason for this is not clear, although it may be attributable to the difference in compatibility of particle size distribution and/or shapes between the different binders.

### 6.7.2 Uniaxial Compressive Strength

Mortar and concrete cubes made using Portland cement as the binding agent develop higher strengths and develop strength faster than cubes mixed with Red Gypsum binders. Red Gypsum Binder A generally developed higher strengths than Red Gypsum Binder B, in some cases 100% higher. For all 3 binders, (aside from a few anomalous results), lower water contents in the mixes generated higher uniaxial compressive strengths.

Without conducting extensive mineralogical analysis it is not possible to explain fully the reasons why the Portland cement samples achieved higher strengths than the samples made with the two Red Gypsum binders. One aspect that will have been influential is the water content of the initial mixes. Standard high strength concretes are usually made with low water contents. For the sake of achieving mixes with reasonable workability it was nessesary to add more water to the Red Gypsum mixes than the Portland cement ones. Also, Binder B mixes required more water to achieve sufficient workability. This increased water content probably accounts for a significant part of the strength difference between the different binder types. If Red Gypsum binders were to be used in the production of paving blocks then in order to maximise strengths water content should be kept to a minimum.

Despite the initial low strengths the cubes made with Red Gypsum binders were strong enough to be taken out of sample moulds and handled after one day curing. This would be important if Red Gypsum paving blocks were to go into production to maintain an efficient production process as once blocks are formed it should be possible for them to be handled and stored without risk of damage.

When ICON sand was substituted for standard building sand in mortar cubes made with Binder A, there was little change in the compressive strength. In mortar cubes made with Binder B the samples containing ICON sand were slightly weaker than samples containing building sand. Conversely, in cubes made with Portland cement where ICON sand was substituted for standard building sand, strengths were higher than those with standard

building sand. Some of these variations can be accounted for by the different water contents used in the various mixes but again a detailed mineralogical investigation is required to fully explain the reasons for the difference in strength. It can be concluded however that ICON sand did not have a significantly detrimental effect on the strength of the cubes and therefore is suitable to be used as a replacement for standard quarried sand in concrete blocks.

The effect of the addition of ochre to the concrete mixes with Portland cement binder unfortunately cannot be properly assessed as an error was made when selecting the water content for the mix. The value selected was too low and this has clearly affected the strengths of the samples. The addition of ochre to cubes with Binder A made little difference to the strengths despite cubes being at similar water contents. In the case of cubes made with Binder B a significant loss in strength was observed, and this again may be due to the mixes having higher water contents in order to maintain workability.

Red Gypsum binders can develop high enough strength to be used to make a low strength concrete as 30MPa 28 days strengths were consistently achieved in mortars and concretes. Also ICON sand can be substituted for standard building sand without causing loss in strength. The use of ICON sand and Red Gypsum in concrete blocks has the potential to reduce the cost of block manufacture and provide a more environmentally sound disposal option than landfill.

### 6.7.3 Ultrasonic Pulse Velocity (UPv)

Samples prepared using Portland cement as a binder developed higher stiffnesses and developed them faster than the Red Gypsum cubes. Generally samples prepared with Binder A exhibited higher stiffness than Binder B (ranging from 15-25% greater). This is consistent with the strength results and again can only be fully explained by a thorough mineralogical investigation; although the water content of the samples will have had a considerable effect on the stiffness of the samples (lower water contents causing greater stiffness) in the same way as it will have had on strength (see Section 6.7.2).

In mortar samples the replacement of building sand with ICON sand raised the stiffness by approx 15% (in the lowest water content samples) where Portland cement was the binder. However, this was not repeated in the Red Gypsum cubes where stiffness remained approximately the same. In concrete samples the replacement of building sand with ICON sand raised the stiffness in both Portland cement and Red Gypsum cubes. These results indicate that in general the replacement of standard building sand with ICON sand increases the strength of the samples, and this increase is probably due to the reduced water content of the mixes that used ICON sand.

The addition of ochre to concrete mixes reduced the stiffness by 10% in Binder A samples and 44% in Binder B samples. No conclusion can be draw from the Portland cement cubes with ochre additions due to variations in water contents.

### 6.7.4 Density

In general cubes made with Portland cement binder were slightly more dense than those made with Red Gypsum binder. Cubes made with Binder A were slightly more dense than those made with Binder B. Since the binder only makes up a small proportion of the mass of the concrete and mortar cubes the reason for the higher densities in the Portland cement samples must be related to the particle packing. It was noted in the workability testing that the Portland cement samples were noticeably more workable than the Red Gypsum cubes at lower water contents. This difference in workability would allow the Portland cement samples to achieve higher particle density at the same compactive effort whilst the cubes were being made. For ease of handling lower density is a desirable property since it make blocks easier to lift and carry, but not if it comes at the expense of durability. Lower density indicates that there will be more air voids in the samples, and hence will be more susceptible to freeze-thaw and wet-dry erosion. Concrete cubes with ochre additions were slightly less dense than cubes without ochre.

### 6.7.5 Colour

In mortar and concrete samples, cubes mixed with Binder A were slightly lighter than those mixed with Binder B. This can be explained by the higher Red Gypsum content of Binder B.

There was no noticeable difference in the colour of mortar samples when ICON sand was used in place of ordinary building sand. This is surprising due to the $TiO_2$ content within the ICON sand, the presence of a white pigment might be expected to make the cubes paler. It could be that there is insufficient $TiO_2$ to make a noticeable difference. However, in the concrete cubes, there was a more consistent colour in the samples made with ICON sand, although the mottling seen in the samples could be a result of oxidation of the iron content of the samples during curing and unrelated to the sand content.

Ochre did not cause a significant change in colour in the cubes made with Red Gypsum – GGBS binders; any the effect the ochre may have had was disguised by the effects of the red gypsum. Ochre did have a significant effect on the colour of the Portland cement cubes turning them from grey to moderate brown.

The colour of the cubes could prove to be significant when it comes to developing the blocks into a marketable product. Concrete paving blocks are frequently dyed to give a more attractive appearance. The dyes used are frequently an expensive additive so if a consistent colour can be achieved using Red Gypsum binders and ICON sand then this could provide a significant cost saving.

### 6.7.6 Conclusions

Red Gypsum binder can develop high enough strengths to be used to make a low strength concrete. 30 MPa 28 day strengths were consistently achieved in mortars and concretes.

Of the two binders, Red Gypsum Binder type A exhibited the most desirable strength and stiffness characteristics.

Both Red Gypsum binders required more water than Portland cement to become workable and developed strength more slowly (consistent with pozzolanic reactions). This is a disadvantage when compared to the Portland cement.

Replacing standard building sand with ICON sand in concrete and mortar mixes had little impact upon strength and stiffness characteristics. ICON sand would appear to be suitable for use in concretes provided leachate standards are also passed.

Ochre appears to have little impact upon strength and stiffness of mortar and concrete cubes. The ochre did not have a significant effect on the colour of the Red Gypsum cubes, but it is possible that it may be used as a dye in Portland cement blocks.

Cubes made using Red Gypsum binders had generally lower densities than those made with Portland cement and should therefore be easy to handle. However, this does indicate that there may be durability issues for blocks made with Red Gypsum binders.

Without conducting mineralogical analysis the reasons for the differences in strength of the various mixes/water contents/binders cannot be fully understood but can almost certainly be attributed varying formations of standard hydrated cement minerals and etteringite. Mineralogical testing conducting in other parts of this thesis suggests that this is the case.

### 6.8 Recommendations

The aim of this programme was to test a variety of Red Gypsum / GGBS concrete mixes and compare their strength and strength development with Portland cement mixes in order to see if there is the potential for Red Gypsum binders to be used in the production of paving blocks. Whilst the programme has achieved this, there is a considerable amount of further work required before Red Gypsum binders can be used in mass concrete and paving

block applications. This programme also attempted to investigate many variables (type of binder, type of sand, mortars and concretes, additions of ochre, quantities of water), which has generated a lot of data across a range aspects of the mixes, but has meant that compromises have had to be made with regard to the number of cubes made with each mix.

Future testing programmes involving the development of Red Gypsum paving blocks should either be much larger, or concentrate on a smaller number of variables. The development of Red Gypsum binders for use in this application would provide a suitable topic for a PhD programme all by itself!

In future programmes, further work should be conducted on the composition of the binder to see if one can be developed that achieves faster hardening whilst at the same time still using a significant proportion of Red Gypsum.

When making the cubes it would be beneficial if a greater number of water contents were tested so that the strength – water content relationship of various mixes can be more fully understood. When doing this, more than one cube should be made (the larger the number the better) but at least 3 cubes of each water content should be made for each curing period.

The durability of the cured cubes should be tested in accordance with British Standards (BS1881).

Mineralogical analysis should be conducted on the cured samples. Because of the nature of the hydrated cement minerals likely to be present XRD analysis is unlikely to be useful on its own. This sort of test should be augmented by SEM analysis conducted on polished sections and unpolished broken sections, so that the interaction between the cement and the aggregates in the concrete can be properly observed as well as being able to identify the morphology of any hydrated cement minerals present.

Once the above investigations have been done, research should continue using BS EN 1338:2003, so that Red Gypsum paving blocks can be developed. It would have been desirable to use these standards in this testing programme but at the time the University did not have the necessary equipment to carry out the tests and it was felt that cube testing etc should be used to find a small number of suitable mixes which could then be tested in a commercial laboratory. BS EN 1338 includes tests to assess many important characteristics of paving blocks including tensile strength, abrasion resistance, slip-skid resistance, fire performance, etc and so provides a comprehensive assessment of blocks made commercially.

In addition to the paving block research, it would be beneficial to make an assessment of Red Gypsum binders in mass concrete application such as trench / pad foundations etc, as this could provide a large market in the long term.

# Chapter 7

# CTRL Field Trial Results

# 7. CTRL Field Trial

This chapter is based on a paper published in the Quarterly Journal of Engineering Geology and Hydrogeology, **Hughes, P. and Glendinning S. (2004)**. *Deep dry mix ground improvement of a soft peaty clay using blast furnace slag and Red Gypsum*, QJEG and H, Vol 37, pp 205-216. A copy of the Paper and other papers based on the research presented in this thesis is contained in Appendix E. Because this section of study involved fieldwork rather than laboratory work it was decided to present the methodology within this chapter, separate to the methodology for rest of the study which is detailed in Chapter 3. The tests presented in this chapter were conducted by several different people, where this was not the author of this thesis it is stated in the relevant section.

## 7.1 Introduction

Deep in-situ soil improvement using the dry mix technique has been pioneered in Scandinavia and Japan where very soft soils of high water content are prevalent (Ahnberg et al, 1995 a and b; Okumura, 1997). The process may be achieved by a variety of methods. One example involves rotating a mixing tool into the ground to the required depth of treatment. Once this has been achieved the rotation is reversed and the tool is withdrawn whilst binder is pumped by compressed air through apertures in the tool, mixing binder with the soil. Because of the orientation of the fins, this process achieves a degree of compaction through the length of the column. The process is illustrated in Figure 7.1.

The technique has not been used in the UK until recently due primarily to the prevalence of stiff overconsolidated soils and the lack of UK- based contractors with experience of the dry soil mix process. However, the deep dry mixing technique was used in 2001 to treat approximately 5m of soft clay and peat during the Channel Tunnel Rail Link Contract 440 (CTRL 440) located near the village of Sandling in Kent, South East England. The contract involved treating an area of 4250m$^2$ using columns with a 91% area coverage. The RLE contract specification required the columns of treated ground to achieve a minimum undrained shear strength of 100kPa and a minimum Young's Modulus of 10 MPa (Hanson et al, 2001), within 28 days.

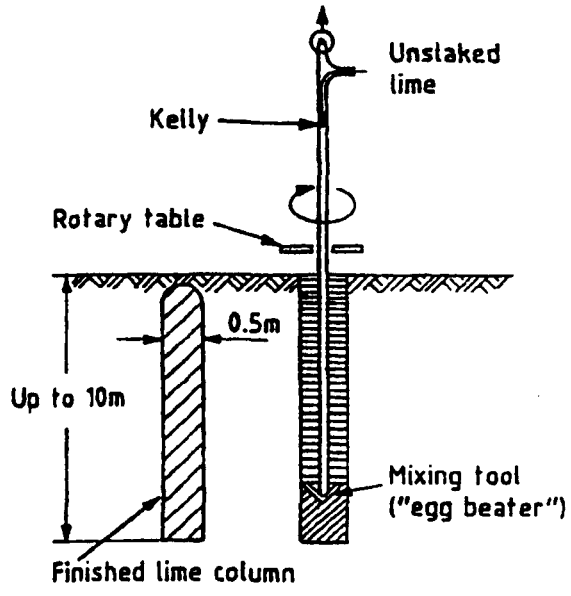



Figure 7.1 An illustration of the 'dry-mix' process (after Broms and Bomen, 1979)

The majority of the cost for this type of stabilisation is considered to be the price of the binder used. The treatment described above required 3000 tonnes of cement at £25 per tonnes to treat 21000m$^3$ of soil with a binder concentration of 200kg per cubic metre of soil. Therefore recent research has concentrated on reducing the cost of the binders through experimenting with the whole or partial substitution of waste or marginal materials for cement. Potential replacements have include Pulverised Fuel Ash (PFA), Lime, and Ground Granulated Blast Furnace Slag (GGBS).

As has already been discussed in Chapter 2 Beretka et al, (1996) have identified the potential for the use of gypsum-based industrial bi-products as alternative sources of cement. Chapters 4, 5, and 6 have demonstrated that when mixed with GGBS Red Gypsum can produce an effective cement replacement. It was therefore decided to trial Red Gypsum – GGBS binders in-situ as a soil mixing binder.

Peat is known to be problematic for cement-based stabilisation techniques as the setting process of cement is retarded by the interaction of the calcium ions and organic matter. Studies have shown that Ground Granulated Blast Furnace Slag

(GGBS) can be effective in the stabilisation of organic soils (Nenad, 1999). Indeed, (Hebib and Farrel. 2003) have identified its use in combination with gypsum as a potential stabiliser for peat-based soils. Results discussed in Chapter 5 have shown that Peat is particularly difficult to treat in the laboratory.

The aim of this field trial was to investigate the potential of using a GGBS-Red Gypsum binder for the improvement of very soft peat *in situ* and to compare its performance with that of OPC. A trial using these alternative binders was conducted adjacent to the ground works for the Channel Tunnel Rail Link. This meant that equipment and material were readily available and a direct comparison of the efficiency of the installation process using the alternative binders to that using Ordinary Portland Cement (OPC) could be made. A further advantage was that although laboratory trials could be conducted in order to assess the most promising binder mixes, the techniques used to mix the peat with the binder in the laboratory could not accurately model in-situ mixing, and therefore could not produce a sufficiently sound case for the practical use of the new binder. Additionally, the disturbance to the peat caused by excavation would cause the laboratory tests to be less representative of in-situ conditions. The advantages were considered to outweigh the possible disadvantages of lack of precise control and uncertainty over exact ground composition with depth. These issues are discussed further in the following sections.

### 7.2 Design Mixes

An initial laboratory testing programme was conducted with the aim of optimising the proportions of Red Gypsum to GGBS and the proportion of binder required to be added to the soil. The methodologies developed in Chapters 4 and 5 were applied in practice. It was also recognised that the addition of a sulphate to a soil as part of a stabilisation process should be approached with caution due to the potential for ettringite (and its subsequent conversion to thaumasite), a material that has potential to swell on contact with water and severely weaken the stabilised material (Mitchell and Dermatas, 1990; Snedker, 1996, and BRE, 2001). The potential for ettringite-thaumasite formation was also investigated.

### 7.2.1 Strength

A laboratory trial was conducted to select the proportions of Red Gypsum and GGBS in this part of the study by technicians at Newcastle University (Anon, 2001). GGBS and Red Gypsum were mixed in varying proportions by mass. Water was then added to achieve a water content similar to that produced when 200kg/m$^3$ of dry binder is mixed with the peat at its natural water content, and tested for strength in undrained triaxial compression. The results of the laboratory tests, although not conclusive indicated that, in these circumstances, the 75:25, GGBS: Red Gypsum mixture produced the highest strength. This was in broad agreement with the results of the binder trial detailed in Chapter 4, although it should be noted that in the trial detailed in Chapter 4 water content was also a variable whereas in this case a standard water content was used throughout. This would account for the differences in strength achieved between the two studies as water content has been shown to have an effect on the final strength of the binder. As the most significant cost savings (the maximum use of Red Gypsum) would be achieved from using the highest possible proportion of Red Gypsum, a combination of 25:75 GGBS: Red Gypsum was also selected for the trial as it produced only marginally weaker material than the 75:25 mix in the laboratory.

Laboratory trails mixing Red Gypsum – GGBS binders with Peat from the site of the field trial were conducted by Simpson , (2001). Bulk samples of peaty clay were taken from the site of the proposed field trial and hand mixed in the laboratory with GGBS – Red Gypsum binders at their natural moisture content in order to determine the proportion of binder required to stabilize the soil. The samples were compacted using a tamping bar to a bulk density of 1.1Mg/m$^3$ within a 38mm diameter mould, transferred to plastic sample tubes, sealed, and allowed to cure at 20 C for 7, 28 and 56 days. The target bulk density was that of the in-situ columns and has been found to be an important parameter in laboratory-based design of dry-mix columns (Ekman and Holmgren, 2002). For comparison similar samples were prepared using Ordinary Portland Cement and cured under the same conditions.

Undrained triaxial compression tests, pH tests and X-ray diffraction analysis were performed on all the samples. These tests concluded that, whilst the modified soil had greater shear strength than the original peat, and increasing the amount of binder increased the shear strength of the samples, there was no apparent increase in strength with curing time. This implied that pozzolanic (cementitious) reactions had not taken place. Soaking of the samples for 24 hours under water prior to testing was found to reduce the measured strength by approximately 25%. On average, the samples mixed with cement were 65% stronger and suffered less strength reduction when soaked than the samples mixed with GGBS - Red Gypsum.

Investigative testing into the possible cause of this problem found that the pH of the peat samples delivered to the laboratory was below 2. This was surprisingly low compared to recorded values of the order of 7 for natural peat in the field (Ekman and Holmgren, 2001). The addition of the GGBS – Red Gypsum binder increased the pH of the peat, with larger quantities of binder producing greater increases in pH. However, pH values rarely exceeded 8, a value which is significantly below the pH required for pozzolanic reactions to take place.

### 7.2.2 Ettringite and Thaumasite Formation

In order to investigate the potential for ettringite-thaumasite formation, a set of samples were cured through a temperature cycle of 20C-4C-20C and subsequently soaked in water. These conditions were considered conducive to ettringite-thaumasite formation based on the work reported by Snedker (1996). X-ray diffraction testing conducted on these samples indicated the presence of the mineral pyrite, however no evidence of ettringite or thaumasite was found. There was no obvious difference between the mineral composition of untreated samples and the samples treated and cured for different lengths of time indicating that curing time had no determinable mineralogical effect.

These tests concluded that using a binder of GGBS and Red Gypsum improved the strength and durability characteristics of the peat. However, the low pH of the peat did not provide the required conditions for pozzolanic (cementitious) reactions to take

place and hence no long-term stability was achieved. Therefore, the most probable reason for the low pH values of the peat was oxidation of the pyrite after exposure to the air. Unfortunately it is not possible to check for the oxidation products of pyrite using XRD as they are amorphous. With no pozzolanic reactions no ettringite or thaumasite could form.

Despite the results, it was considered that the GGBS-Red Gypsum binder had potential for use in-situ where the mixing process meant that the soil was not exposed to the air preventing the oxidizing conditions that caused the low pH values. A selection of binder mixes to be used in the field trial (see Table 1) was designed on the basis of the 'binder-only' test results The proportion of binder used for stabilization using the dry mix process was based on the experience gained during the execution of the CTRL stabilization works. It was noted that the proportions selected were in line with those used by Hebib and Farrel (2003). In addition to the GGBS-Red Gypsum mixes a number of dry mixed columns using Ordinary Portland Cement were installed so that a comparison could be made. The nomenclature in the left – hand column of Table 7.1 will be used throughout the rest of the Chapter to describe the different columns in the trial.

## 7.3 The Field Trial

The regional geology around the location of the trial site has been described by Ekman and Holmgren (2001) and broadly consisted of soft marine deposited sedimentary rocks of various lithification known as the Lower Greensand Formations and the Gault Clay. Two cable percussion boreholes were excavated in proximity to the trial area along with seven trial pits. These demonstrated that the local ground conditions consisted of sands of the Sandgate and Folkstone beds overlain by peaty CLAY/clayey PEAT in turn overlain by a thin layer of topsoil. In-situ testing showed that the peat/clay had a typical undrained shear strength of 10 kPa but contained two very soft layers at approximately 2.2m and 4.2m, with strengths of below 5kPa. Peat recovered from the boreholes and tested in the laboratory had a water content of 327%, a loss on ignition of 55%, and a Von Prost (degree of humification) value of 9.

Table 7.1 Binder mixes and concentrations.

| Mix | Concentration (kg/m$^3$ soil) | Ratio (by mass) |
|---|---|---|
| A/G75/200 | 200 | 75% Red Gypsum, 25% GGBS |
| B/G75/250 | 250 | 75% Red Gypsum, 25% GGBS |
| C/G25/200 | 200 | 25% Red Gypsum, 75% GGBS |
| D/G25/250 | 250 | 25% Red Gypsum, 75% GGBS |
| E/C100/200 | 200 | 100% cement |

A total of 27 columns were installed using the combination of binder concentrations and binder ratios shown in Table 7.1. Powdered Red Gypsum and GGBS were delivered to the site by tanker and mixed in the required proportions in the binder delivery vessel. The columns were then formed in exactly the same way as the OPC columns in the full-scale CTRL works, with similar rates of production being achieved. Once the columns were installed there was a delay of 24 hours before a 1.5m layer of fill surcharge was placed over them. This delay was due to activity on the main site works. Columns on the CTRL route were loaded after a period of four hours as recommended by Ahnberg et al (2001).

### 7.3.1 Testing

The laboratory testing of samples recovered from the field trial, and analysis of the results of in-situ testing was conducted by the author of this thesis at Newcastle University. A combination of post treatment in-situ and laboratory testing was used to assess the effectiveness of the ground improvement compared to that of OPC. The most important parameters for comparing the performance with that of OPC were considered to be: strength, stiffness, rate of strength development, and durability.

Strength and stiffness were assessed in-situ using the Standard Column Penetration Test (SCPT) and in the laboratory using the Quick Undrained Triaxial compression test. The rate of strength development was determined by testing samples after different curing periods. Durability was assessed in the laboratory by soaking samples in water, and by subjecting samples to freeze thaw, and wet and dry cyclic conditions. The effects of these 'environmental' conditions on the subsequent laboratory strength of the columns was also determined. As previous laboratory testing had indicated that pH was key to strength development, the pH of all samples was determined. The mineralogical composition of the stabilised soil, including the potential presence of ettringite and/or thaumasite was investigated using X-ray diffraction.

**In-situ Testing**

Eight columns were tested at 7 and 56 days after installation using the standard column penetration test (SCPT) in which a special two-fined probe was fitted to the end of a standard Cone Penetration Test (CPT) probe (see Figure 7.2). The probe was then pushed down through the centre of the column at a constant rate of 20mm/s using the equipment used to form the columns. The resistance on the probe was recorded continuously and the undrained shear strength (Cu) correlated to 10% of the converted resistance force (Carlsten 1995).

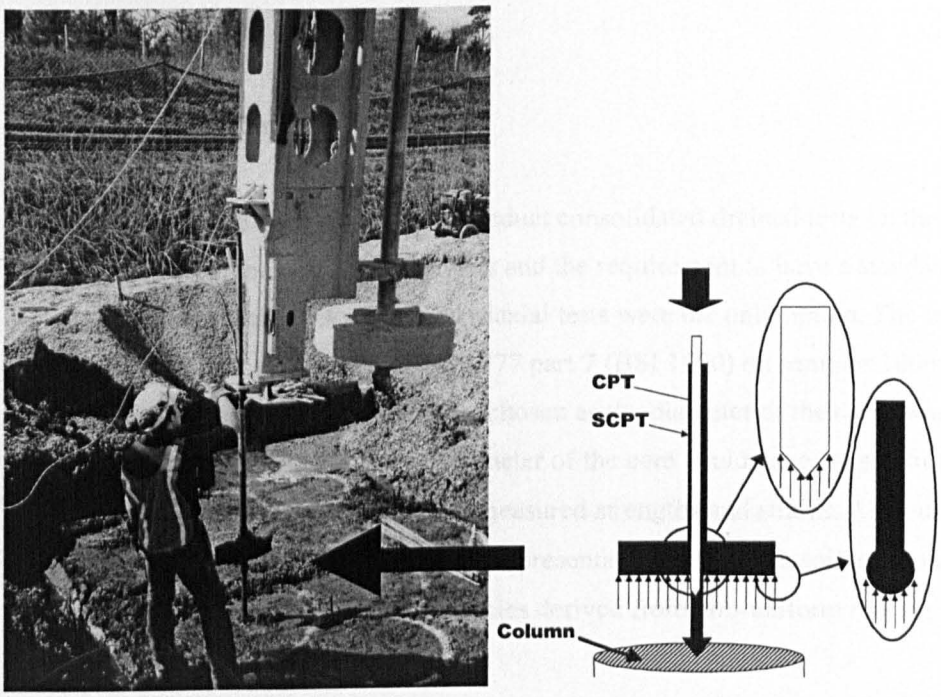



Figure 7.2 In-situ testing equipment.

**Sample Recovery**

Five rotary boreholes were drilled from the platform layer 1.5m above the top of the columns using a TR80 rotary rig with a triple tube core barrel and air mist drilling fluid. Cores of 100mm diameter and 1.5m long were taken from the top of the columns to a depth of 6m. Recovery rates were relatively low: columns C/G25/200, D/G25/250 and E/C100/200 had core recovery rates of between 80 and 88%; columns A/G75/200 and B/G75/250 had rates of 70 and 62% respectively. Cores were stored in sealed plastic tubing in order to protect sample integrity and prevent any potential oxidation. 100mm triaxial sub-samples were taken approximately every 1m (or the closest 200mm long piece of intact core). Once tested for undrained shear strength the samples were retained for pH testing. Freeze thaw, durability and wet/dry tests were performed on additional samples taken from the remaining available intact core.

**Laboratory Testing**

**Triaxial Compression**

Whilst it would have been desirable to conduct consolidated drained tests on the samples recovered, the number of samples and the requirement to have a standard curing time meant that quick undrained triaxial tests were the only option. The tests were performed in accordance with BS 1377 part 7 (BSI 1990) on samples 100mm in diameter and 200mm long. This size was chosen as the diameter of the cores was approximately 100mm. Reducing the diameter of the core could have caused further damage to its structure, thus influencing measured strengths and strains. Also, using larger samples was considered to be more representative of the bulk soil mass and, at least in part, to overcome any inconsistencies derived from non-uniform mixing.

The samples were compressed at a constant rate (in this case 1.5mm/min) whilst under a nominal confining pressure of 100kPa. Whilst it was recognised this did not represent the in-situ stress conditions, it replicated previous laboratory testing procedures. The aim of the testing was to compare the performance of the GGBS-Red Gypsum columns with cement columns, not to produce design strengths, so consistency was required.

As the axial load was applied, the sample suffered continuous deformation. Throughout the tests, until the sample failed, readings of stress and strain were made at regular intervals. If the sample continued to deform without failing, then the value of stress at 20% strain was recorded as the failure stress. In some cases the samples did not fail before the maximum load of the proving ring had been reached. These samples were cored down to 38mm and the tests repeated at a 0.5mm/s strain rate.

**pH and Mineralogical Testing**

The pH of the samples was determined by the method outlined in BS 1377 part 3 using a Jenway pH meter (model 3150), giving a direct reading of the pH value of the soil suspension in water. Samples were prepared by mixing a 30g air-dried sample with 75ml of distilled water for a period of at least 8 hours. Mineralogical testing

consisted of X-ray diffraction, Scanning Electron Microscopy (SEM) and Petrographic Microscope analysis. X-ray diffraction was performed on 2g of air dried sample, ground to no greater than 10 microns in size and compressed onto a slide for analysis. Petrographic analysis was conducted on standard thin sections, SEM was conducted on polished sections.

**Freeze Thaw and Wet -Dry Testing**

The freeze thaw tests conducted in this study have been based on the ASTM procedure D560. However, due to the nature of the samples recovered from the field trial it was not possible to create samples of the specified dimensions (101.6 +- 0.41mm diameter, 116.43mm in length) so 50mm cubes were used instead. In accordance with ASTM D560, these cubes were exposed to 24 cycles of freezing at – 10 degrees Celsius, and thawing at 21 degrees Celsius. It was decided not to brush the samples after each cycle as specified in the standard as the small and angular nature of the samples would have made them far more susceptible to damage than samples of the dimensions set out in the standard. In addition to the cube tests, seven 38mm triaxial samples were exposed to freezing and thawing cycles in order to assess the effect of freeze-thaw on shear strength.

The wet and dry tests were loosely based on ASTM procedure D559. The dimensions of the samples were 38mm diameter, 78mm long, again to allow the effects of wet-dry cycles on undrained shear strength to be determined. The drying cycle was carried out at 25C because the Red Gypsum used contained structural water, which would be driven off at temperatures over 60C. As a temperature of over 70C is highly unlikely to occur at depths much below the ground surface this was considered to be a well-founded amendment to the Standard procedure. Samples were tested in undrained triaxial compression after 4 wetting and drying cycles.

## 7.4 Results

### 7.4.1 In-situ Testing Results

The response of the GGBS-Red Gypsum columns to the in-situ testing was typified by a peak in shear strength at approximately 0.4m depth, dropping to a minimum value at around 1.9m depth and steadily increasing again to between 4.0 and 4.5m depth where the tests terminated (see Figure 7.3).

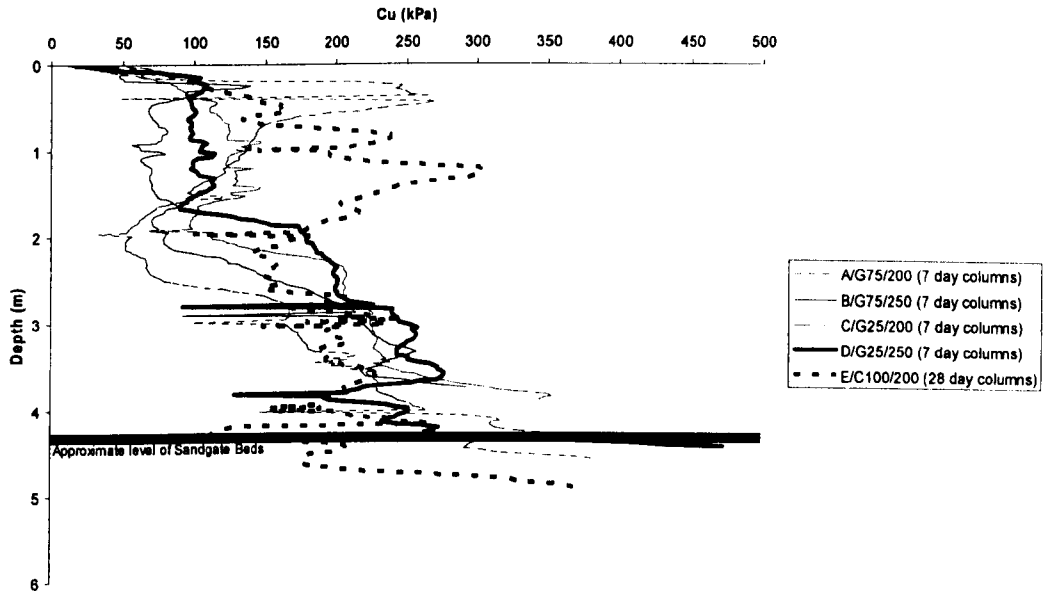



Figure 7.3 Undrained shear strength of each column type with depth after 7 days curing (derived from in-situ testing results)

The pure cement columns exhibited higher shear strengths than the GGBS-Red Gypsum columns. Generally the shear strength exhibited was higher in the columns with a higher concentration of binder for both GGBS and cement columns. However, in the case of the GGBS – Red Gypsum columns, it was difficult establish which binder ratio was more effective, with no mix showing a consistently higher strength for the entire length of the column. The considerable variation in shear strength was probably attributable to varying initial ground conditions and, possibly, mixing efficiency. As stated earlier, the boreholes encountered softer ground in parts of the peat layer, and these weaker zones were likely to occur at differing depths spatially across the trial area or may have been absent in some parts altogether.

The fact that it was not possible to perform SCPT tests in the GGBS-Red Gypsum columns after 56 days shows that the shear strength continued to increase in the period between 7 and 56 days which is consistent with the development of pozzolanic reactions.

The GGBS-Red Gypsum columns showed considerably greater shear strengths than the samples tested in the initial laboratory testing thus confirming that the lack of significant improvement in shear strength was probably caused by samples oxidizing before testing, reducing in pH, thus preventing pozzolanic reactions from taking place.

The GGBS-Red Gypsum columns showed a much wider variation in shear strength than the pure cement columns, with shear strengths dropping to 50 kPa in some places. However, as previously stated, this effect may be due to varying initial ground conditions. The trial columns were installed in a different area of the site from the main works where ground conditions would have been different. This coupled with the fact that testing was conducted after a longer curing period (56 days as opposed to 21 and 30 days) from the time of the installation makes comparison difficult.

### 7.4.2 Laboratory Testing Results

**Strength**

It can be seen from Figure 4 that mix A/G75/200 and B/G75/250 exhibited significantly lower shear strengths than the other samples tested, with average undrained shear strengths of 104 and 67kPa and minimum and maximum strengths of 35 and 230 kPa and 23 and 94 kPa respectively. Mixes C/G25/200 and E/C100/200 showed considerable variation, but had approximately the same average shear strengths of 670 and 923 kPa. Mix D/G25/250 showed the highest shear strength, with an average strength of 1946 kPa and minimum and maximum strengths of 1511 and 3114 kPa respectively.

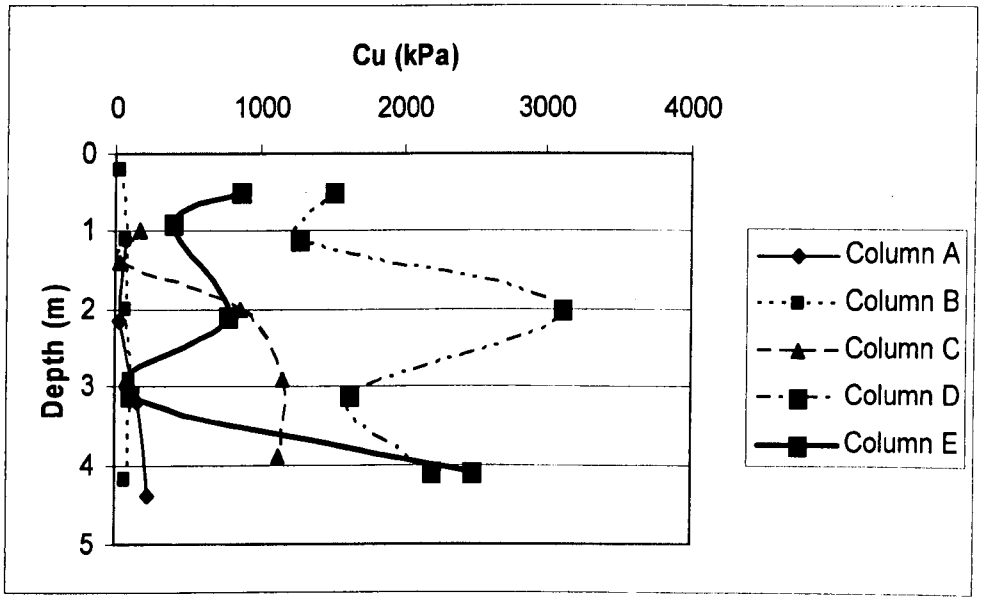



Figure 7.4 Results of laboratory strength testing plotted against depth

All mixes exhibited lower shear strength values between 0.9 and 2.1m, corresponding with results from in-situ testing and attributed to natural zones of weakness within the peat layer.

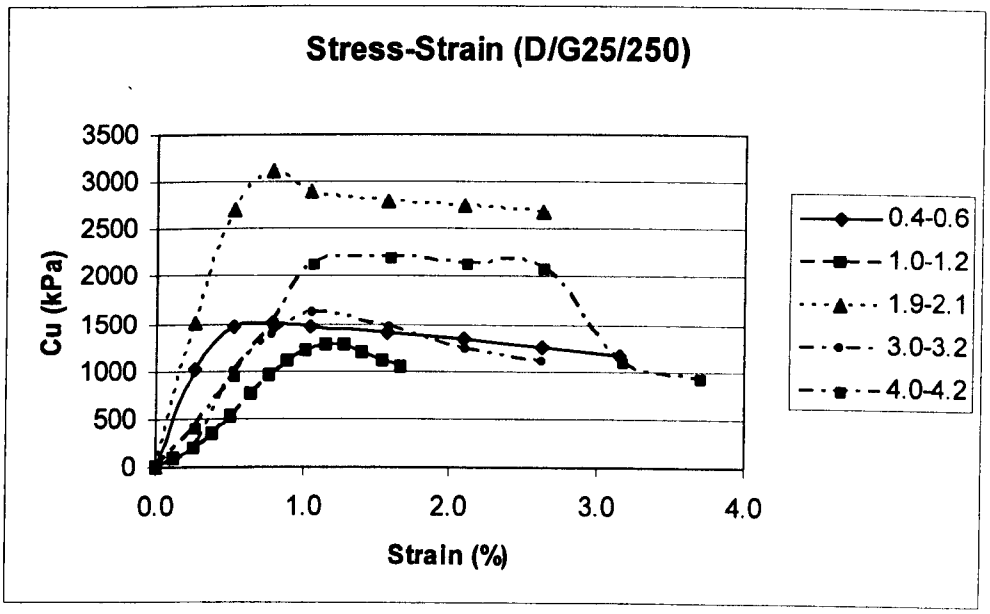



Figure 7.5 Stress-strain characteristics for Column D.

Figure 7.5 shows the stress-strain results from column D/G25/250, as noted previously the lowest shear strength is exhibited by the sample taken from 1m depth. It should also be noted that peak strengths were reached at strains typically lower than 1%. However, once the peak strains were reached the post peak strengths remained relatively high, considerably above the 100kPa CTRL specification.

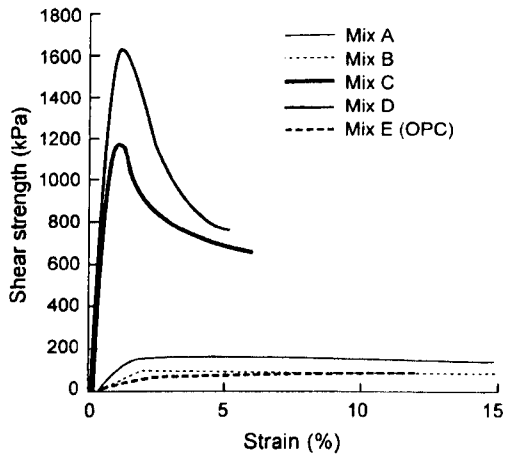



Figure 7.6 Stress-strain behaviour of each column mix.

Figure 7.6 shows stress strain plots from all columns on samples taken from 3m depth. It can be seen clearly that the samples with greater strengths and stiffnesses reached peak strengths at much lower strains than the weaker samples. Despite this the two stronger samples (C and D) maintain a strength of over 600kPa past 5% strain.

**Stiffness and strain to failure**

Table 7.2 shows that mix A/G75/200 and B/G75/250 also exhibited significantly lower Young's Modulii, averaging 8.8 MPa and 3.7 MPa respectively. The other mixes exhibited values between 70 and 230 MPa, with mix D/G25/250 showing the highest value. It should be noted that there was considerable variation in the values of Young's Modulus for each individual mix.

Table 7.2. Summary of Strengths and Stiffness from Laboratory Tests.

| Column | Average Undrained Shear Strength (kPa) | Average Young's Modulus (MPa) | E/Cu |
|---|---|---|---|
| A/G75/200 | 81 | 9 | 111 |
| B/G75/250 | 42 | 4 | 95 |
| C/G25/200 | 586 | 123 | 210 |
| D/G25/250 | 1166 | 228 | 196 |
| E/C100/200 | 357 | 71 | 199 |

Mixes A/G75/200 and B/G75/250 had the highest average strains at failure with averages of 10.5% and 7.5% respectively. Mixes C/G25/200 and E/C100/200 averaged 6.0 and 4.0 % respectively, while the average failure strain for mix D/G25/250 was 1.0%.

**Strength Development**

It can be seen in Figure 7.7 that the laboratory shear strength of mixes C/G25/200, D/G25/250 and E/C100/200 are higher than those measured during in-situ testing (increases of 360%, 1240% and 400% respectively). It would appear, therefore, that strength continued to develop in the period between SCPT testing and recovery of the samples. In the other columns the shear strength remained similar, with the exception of B/G75/250 where the average undrained shear strength actually reduced by 50%, although this could be attributed to damage inflicted during sample recovery or transport.

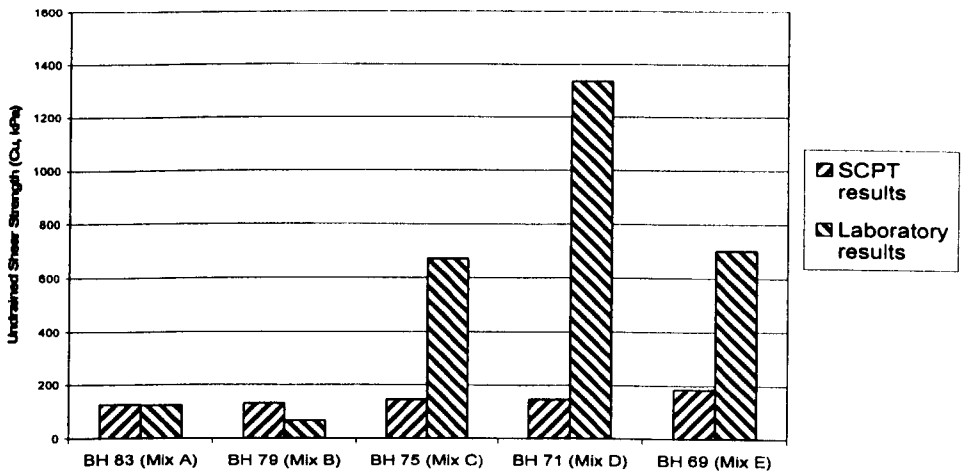



Figure 7.7 Comparison of laboratory and in-situ strength results.

**Durability Testing**

**Freeze Thaw and Wet/Dry Testing**

As can be seen from Figure 7.8 and Table 7.3 there was only a limited difference in the shear strength between the control samples (those samples not subjected to freeze-thaw or wet-dry) from C/G25/200 and D/G25/250 and the samples exposed to cycles of freezing and thawing, and wetting and drying. Differences as small as these could have been caused by variations in the quality and consistency of the initial samples. Triaxial testing has shown that there was considerable variation in the strengths of all samples. Figure 7.9 shows the peak undrained shear strengths from 38mm quick undrained triaxial samples when soaked and un-soaked. Two of the four samples tested (D/G25/250 @ 0.4-0.6m and E/C100/200 @ 4.0-4.2) show little change in strength due to soaking. However samples D/G25/250 @ 3.0-3.2m and D/G25/250 @ 4.0-4.2m increased in shear strength when soaked, in the case of the latter by around 100%, but this again could be explained by natural sample variation.

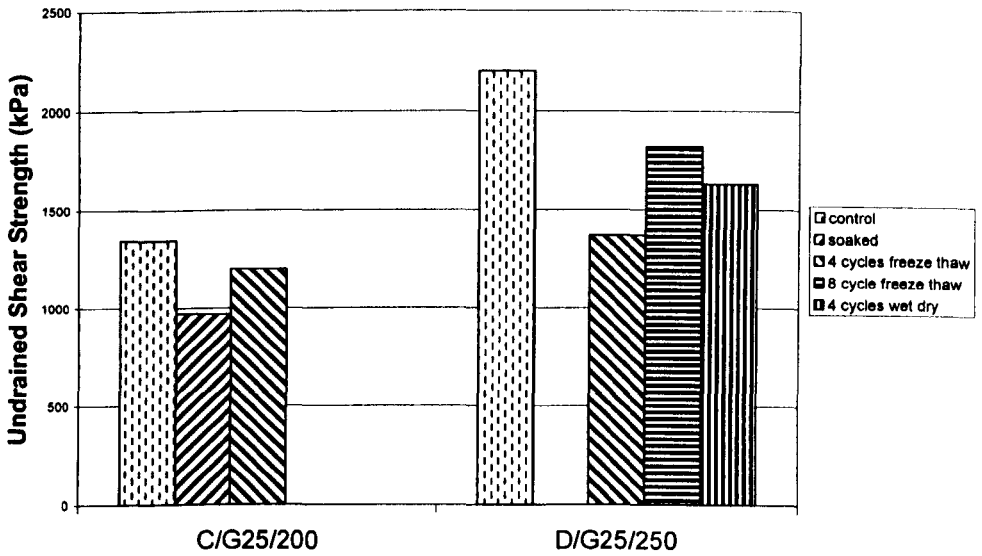



Figure 7.8 Undrained shear strength of column C and D after subjection to freeze-thaw and/or wet-dry cycles.

Because of the low strengths of samples from A/G75/200 and B/G75/250 it was not possible to make cube samples from the core so it was only possible to test mixes

C/G25/200, D/G25/250 and E/C100/200. Two samples were taken from each mix. Where possible one was taken from as close to 2m depth as possible in an attempt to test samples from the weaker layer noted in the borehole logs.

Table 7.3. Results of freeze thaw and wet dry triaxial test.

| Sample | Undrained Shear Strength (kPa) | |
|---|---|---|
| | C/G25/200 | D/G25/250 |
| Control | 1341 | 2198 |
| Soaked | 968 | Not tested |
| 4 cycles freeze thaw | 1198 | 1371 |
| 8 cycles freeze thaw | Not tested | 1821 |
| 4 cycles wet-dry | Not tested | 1633 |

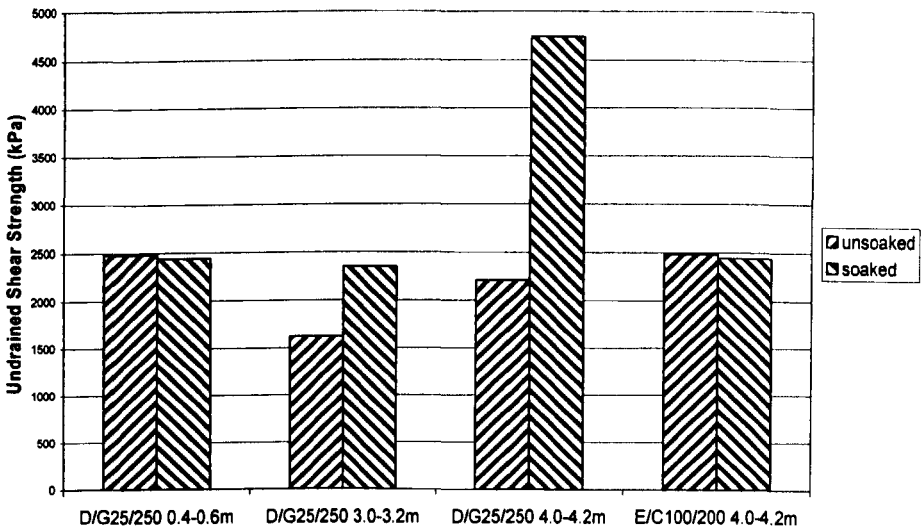



Figure 7.9 Undrained shear strength of column D and E after subjection to soaking.

All samples remained intact for the first 10 freeze thaw cycles. Over the course of the first 10 cycles of freezing and thawing samples C/G25/200 @ 2.0m, C/G25/200 @ 3.0m, D/G25/250 @ 3.3m, and E/C100/200 @2.0m lost little (less than 4%) of their mass, indicating that they were durable under these conditions, as shown on Table 7.4.

Table 7.4. Results of freeze thaw durability tests (first 10 cycles, mass measured at end of thaw).

| Cycles | C/G25/200 @ 2.0m | C/G25/200 @ 3.0m | D/G25/250 @ 1.8m | D/G25/250 @ 3.3m | E/C100/20 0 @ 2.0m | E/C100/20 0 @ 3.0m |
|---|---|---|---|---|---|---|
| | Loss of mass (%) | | | | | |
| 2 | -0.8 | -0.8 | -0.5 | -0.6 | -0.8 | -3.4 |
| 4 | -1.3 | -1.6 | +0.1 | -0.6 | -1.0 | -4.8 |
| 6 | -1.5 | -2.4 | +0.9 | -0.6 | -0.5 | -9.1 |
| 8 | -1.9 | -3.3 | +1.8 | -0.5 | -0.6 | -21.4 |
| 10 | -2.0 | -3.5 | Sample destroyed | -0.5 | -0.5 | -31.9 |

Sample E/C100/200 @ 3.0m began to lose mass after the first cycle and continued to degrade until it had lost more than 30% of its mass by the 10th cycle. Sample D/G25/250 @ 1.8m gained mass steadily (attributed to it taking on more water) until cycle number 8, at which point its mass had increased to 1.8% more than its initial value (actually 5% when taken after the end of the freeze cycle). The sample then split into three parts and was effectively destroyed. In the majority of cases the samples proved to be durable against the action of freeze-thaw; the samples of mix D/G25/200 in particular showed little effect. All samples that remained intact after the $10^{th}$ cycle were tested to the full 24 cycles. For sample E/C100/200 @ 2.0m, between 10 and 15 cycles the mass of the remaining sample did not change significantly but on the $16^{th}$ cycle it disintegrated. During the remainder of the test the mass of sample D/G25/250 @3.3m remained constant whereas samples C/G25/200 @ 2.0m and 3.0m suffered significant reduction in mass although they did not totally disintegrate.

During the freeze thaw cycles conducted on the cube samples the dimensions of the cubes were measured at regular intervals. Through this it was possible to gain an indication of the shrink and swell of the modified soil. It was found that the maximum swell occurred in sample E/C100/200 @ 3.0m (0.9%); samples D/G25/250 @ 1.8m and 3.3m exhibited swells of 0.7 and 0.5% respectively, but both samples from mix C/G25/200 had maximum swells of 0.3%.

### 7.4.3 Mineralogical Testing

All samples exhibited pH values between 8.5 and 12.1. Samples from mix A/G75/200 showed the lowest values of pH at 8.5. In no case did the pH values fall to the values exhibited by the original laboratory samples.

None of the 27 samples tested in XRD exhibited any evidence of the presence of ettringite or thaumasite. There was also no evidence of the presence of Red Gypsum. Unfortunately due to their amorphous nature, XRD is not suited to investigating the presence of hydrated cement products.

Petrographic analysis showed that sections taken from the weaker mixes, A and B, contained visibly more organic material than those taken from mixes D, C and E. Also noted was the presence of numerous sand grains. Again, no evidence of ettringite or thaumasite was found.

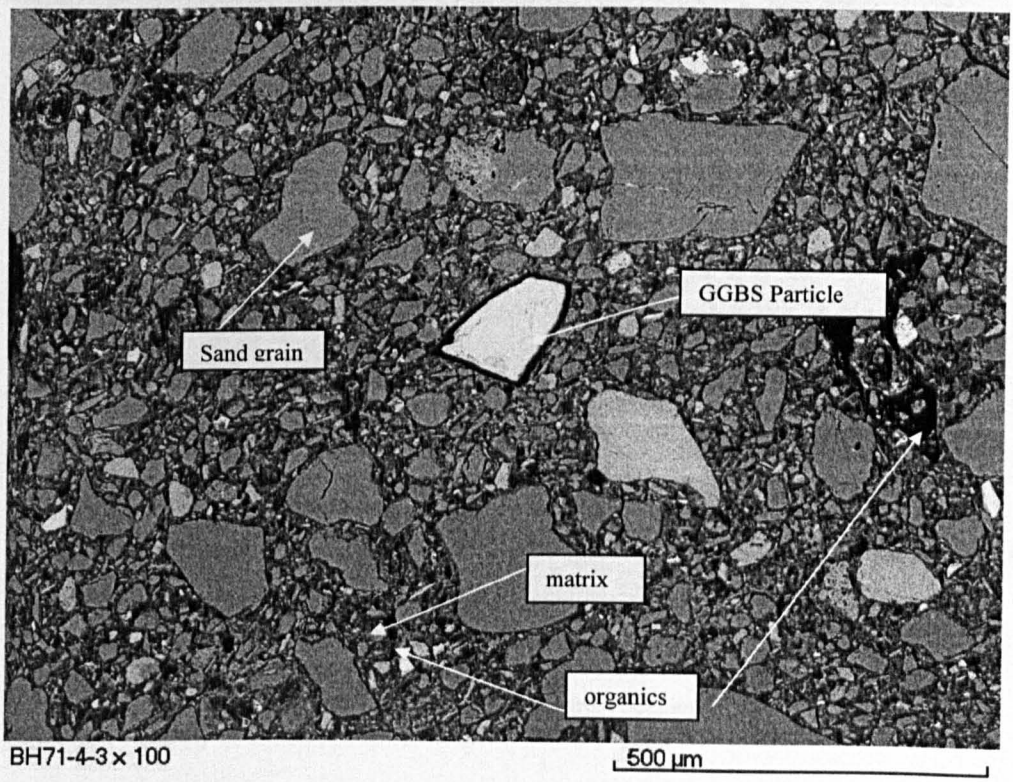



Figure 7.10 Polished section from column D.

Polished sections were made from samples taken only from columns D and E. The slides were examined using a SEM (see figures 7.10 and 7.11). Examination showed the specimens contained very few void spaces, implying that those initially present had been filled with hydrated cement products. Only small areas of organic material were seen to be present, and the presence of some sand grains was noted. Elemental analysis was conducted on several grains observed on the slides, the results of which confirmed the presence of silica grains, particles of un-hydrated GGBS and Red Gypsum (in mix D), particles of un-hydrated OPC (in mix E), and amorphous hydrated cement products in the sample matrix. Again no evidence was found for the formation of ettringite or thaumasite. Elemental analysis showed the needle shaped crystals present (see Figures 7.10 and 7.11) to be Red Gypsum. This indicates that the XRD equipment was not sufficiently sensitive to pick up the Red Gypsum in the bulk material.

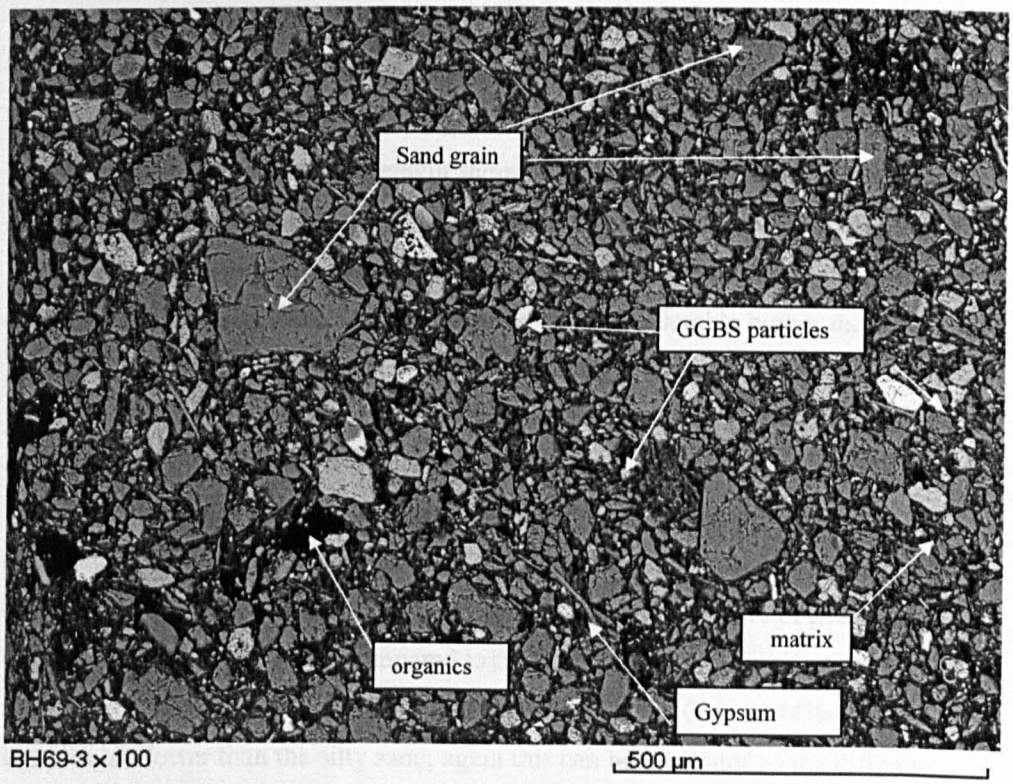



Figure 7.11 Polished section from column E.

## 7.5 Discussion

The average strengths measured *in situ* after 7 days of formation were of similar magnitude for all the columns tested, particularly when viewed in the context of the variability of the original ground conditions.

The laboratory testing indicated that C/G25/200 reached the same average strength as that of the cement column mixed at the same binder-soil proportion. However the same GGBS-Red Gypsum binder achieved an average strength of 200% of the OPC column when the binder proportion was increased to 250kg/m$^3$. D/G25/250 and C/G25/200 both exhibited higher Young's Modulii than the OPC column, in the case of D/G25/250 nearly three times higher. The strain to failure of D/G25/250 was however, only 25% of that of the cement column, whilst the lower proportion of binder produced an increase to a strain at failure of 33%. Looking at the ratio of Young's modulus to undrained shear strength (E/Cu), Table 7.2, the values for two columns containing the higher proportion of gypsum (A and B) were approximately half that for the higher proportion of gypsum (C and D). The values for C and D were very similar to that of the pure cement samples. Despite the relatively low strains to failure exhibited by the stronger samples post peak strengths remain higher than the 100 kPa specified for the CTRL project, in most cases several times higher. The strengths exhibited by the CTRL samples are very considerable higher than the strengths of the peat samples tested in the laboratory discussed in chapter 5. This can be accounted for by the fact that the CTRL samples contained significant quantities of clay and sand material whilst the laboratory samples were almost exclusively organic. Also the curing of the CTRL samples occurred at depth under considerably higher confining pressures than were achieved in the laboratory, as has already been discussed the confining pressure has a major effect on the strength of peat soils. When the CTRL samples are compared to the other soils tested in chapter 5 the CTRL samples achieved higher strengths than the London Clay and Glacial Till samples but lower than the silty sand; again this can be attributed to the different composition of the soils.

Previous studies (Hebib and Farrel, 2003) have noted an increase in stiffness with an increase in confining pressure. Analysis of the results from the GGBS-Red Gypsum

improved columns did not show such a relationship. This can be explained however, as the variability in the initial ground conditions having much greater control over the final properties of the stabilized peat, and initially weaker zones were to be found to correspond with increasing depth.

As with the samples of other soils mixed in the laboratory there was strong evidence that pH was a controlling factor over the strength of the GGBS-Red Gypsum columns. Overall the results from the pH testing did not indicate that samples with higher pH values produced higher shear strengths, but that high shear strengths were not reached unless the pH was above 10.5 to 11. This is illustrated in Figure 7.12. This should not be considered surprising as a pH above 10.5 is required to bring alumina and silica into solution in order to produce cementitious

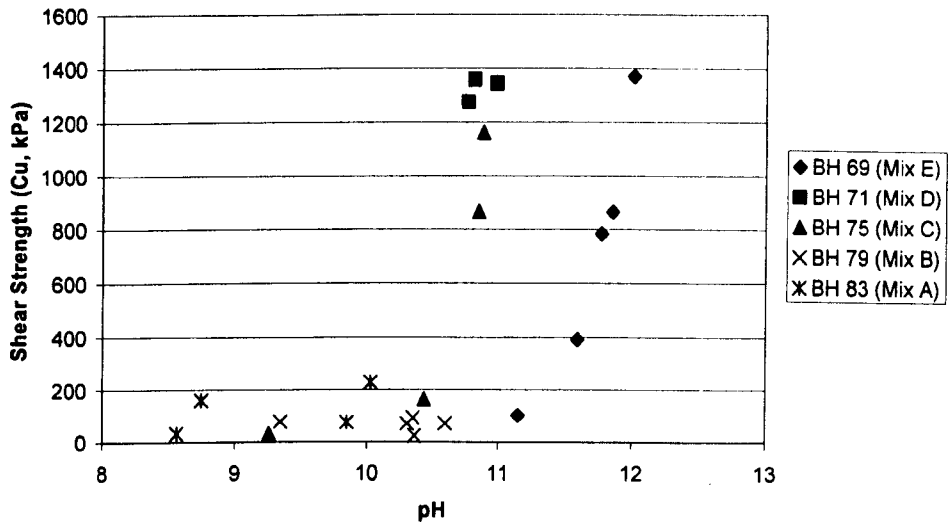



Figure 7.12 Comparison of undrained shear strength and pH of sample.

compounds (Eades and Grim, 1966) and thus produce appropriate conditions for pozzolanic reactions to occur. Kuno et al (1989) also noted that increased levels of humic acid in organic soils influenced the soil improvement factor. Mixes with the higher proportion of Red Gypsum A/G75/200 and B/G75/250 had pH values below this range, and they were also the weakest samples to be tested. As discovered with the initial laboratory testing, if there is a possibility for oxidising reactions to reduce the pH of soil samples, these must be stored in air-tight containers prior to testing. This is particularly important in the process of laboratory-based design of in-situ

mixing of cementitious binders. The extremely high pH of cement means that this is less important for OPC columns, although it is possible that it may have an effect on the calculated binder concentrations. The effect of pH on the initial laboratory samples has meant that the reduction of shear strength values (as recomended by Hebib and Farrel (2003) and EuroSoilStab (2001)) of laboratory results was inappropriate in this case. Whilst it is recognised that strength reduction factors are important in most cases as they take into account, for example, mix energy and efficiency between the laboratory and the field, it is important that they are not applied without due regard for any other difference between laboratory and field conditions which may be in operation.

Soaking did not have a significantly detrimental effect on the undrained shear strength of column D/G25/250 at any depth tested and compares well to the cement column. The cause of the increase in strength at 4.0-4.2m depth has not yet been identified, but is accompanied by an increase in mass (observed in the cube test). As the cube sample increased steadily in mass and then suddenly split into 3 pieces, the possibility of ettringite formation was considered, the needle-like crystals at first increasing the strength, with subsequent swell ultimately reducing strength. However, neither visual observation of the failed sample nor X-ray diffraction results supported this hypothesis.

It would seem that freezing and thawing / wetting and drying do not have a significant impact on the undrained shear strength of the treated peat, but it was not possible to make a comparison with the performance of the OPC columns due to lack of intact samples available for testing.

The mineralogical testing results strongly indicate that pozzolanic reactions have taken place in the GGBS-Red Gypsum and cement mixes and also that ettringite and thaumasite were not present in the samples tested. This contrasts with the samples tested in the laboratory described in Chapter 5 which did contain etteringite this maybe due to the mineral content of the in-situ soil or more likely that some etteringite was present in the soil but that some ettringite was present but quantities were such that it was not picked up in the CTRL samples tested. Hebib and Farrel (2003) noted in their conclusions on testing performed on cementitiously stabilised

peat that cement particles fill the large void spaces that are characteristic of such soils, but that no interaction between hydrated cement products and the organic material could be observed. The samples analysed could best be described as a matrix of GGBS, Red Gypsum, sand, hydrated cement products and organics and it was probable that the organics present had not interacted with the cement. It was noticeable that samples containing larger percentages of organic material also exhibited much lower shear strengths, the strength of the matrix clearly reduced by increased amounts of organic material.

The SEM analysis conducted on the field trial samples revealed the initial soil to be a mixture of clay and sand with variable, but relatively small quantities of organic material and no voids. Therefore the soil could have better been described as a peaty CLAY than PEAT or clayey PEAT. The reason for the discrepancy between this and the available site investigation data could be explained by the fact that boreholes and trial pits were positioned by contractors to obtain best quality information for the CTRL route rather than the field trial. Lateral variation in soil strata to this degree, over these distances is not uncommon. It is an unfortunate fact that in projects of this type where field trials are appended onto large commercial projects that the practicalities of the commercial project must take priority.

### 7.6 Conclusions

From the process of conducting the field trial and subsequent in-situ and laboratory investigations described above, the following conclusions were drawn:

A GGBS-Red Gypsum binder can be substituted for OPC in the dry mix process without any modifications to existing plant and equipment or a reduction in the efficiency of the installation process. No extra operator training or personal protective equipment is required.

From considerations of strength and durability and economics, the most effective binder in this case was considered to be mix C (25% Red Gypsum, added at 200 $kg/m^3$).

The average 7-day strengths of GGBS-Red Gypsum columns were similar to those achieved in the OPC columns. Longer term strengths were found to be in excess of the OPC columns when the proportion of binder was increased from 200 kg/m$^3$ to 250 kg/m$^3$. The increase in the proportion of the binder reduced the strain to failure, but post peak strengths remained in excess of the design specification. Hence the alternative binder was at least as effective as OPC as a stabilising agent for peaty CLAY.

From the tests performed during this study, the strength of GGBS-Red Gypsum columns tested did not appear to be significantly detrimentally affected by soaking, freeze-thaw or wet-dry cycles so it may be argued that the columns are reasonably durable. Again, the alternative binder produced columns that were at least as durable as the cement columns under the same conditions. However, only a limited number of samples could be tested and further testing of the long-term strength characteristics is recommended.

Mineralogical investigations have shown that the minerals formed in the samples from the GGBS – Red Gypsum columns were of the type formed by pozzolanic reactions. It can therefore be reasonably concluded that pozzolanic reactions between the GGBS and the Red Gypsum were taking place. This was further evidenced by the observed pH dependence of the strength of the GGBS-Red Gypsum columns.

Again pH would appear to be a critical factor in the effectiveness of the Red Gypsum – GGBS binder. It is recommended that, if there is a possibility for oxidising reactions to reduce the pH of soil samples, these must be stored in air-tight containers prior to initial design mix testing. Furthermore, if there is any doubt over the pH of the soil-binder mixes this should be adjusted (using a small addition of lime) during the treatment process.

# Chapter 8

# Thesis Discussion and Conclusions

# 8. Discussion, Conclusions and Recommendations

The results from each of the four testing programmes have been discussed within the individual results chapter (4-7). As the results have already been discussed in detail earlier in the thesis to avoid repetition in this chapter early discussions are summarised and combined to enable critical themes of the research to be addressed.

## 8.1 Summary of Individual Chapter Discussions

### Binder Development

In the binder development chapter it was identified that by far the most effective combination tested was Red Gypsum – GGBS. The Red Gypsum – Steel Slag combination also exhibited strength gains but not as large as Red Gypsum – GGBS.

Two stages of Unconfined Compressive Strength tests were conducted in order to optimise the proportions of Red Gypsum and GGBS in the binder, as well as optimising the quantity of water to be used within the samples. These results indicated that the optimum combination was 10% Red Gypsum, 90% GGBS, although it was also shown that a 50:50 mix also achieved high strengths.

XRD mineralogical analysis conducted upon the samples indicated that ettringite was present with the cured Red Gypsum – GGBS binders but that thaumasite was not. SEM analysis indicated the presence of di-calcium silicate. This indicates that pozzolanic reactions had taken place within the binder and that it was this that was responsible for the increase in strength.

**Laboratory Soil Mixing**

In the laboratory soil mixing test programme it was found that a 50:50 Red Gypsum – GGBS binder combination was effective to varying degrees when mixed with a number of soils. It was identified that the primary reason for this variation was the mineralogy of the soils, particularly in respect of how this affected the pH of the soil binder mixture. Achieving a soil-binder pH of above 10.5 was critical in developing high strengths. It was found whilst initially exhibiting high pH, some soils then reduced in pH. One mechanism that could be responsible has been identified as oxidation of sulphate within the soil. This is supported by sulphate testing conducted on samples of untreated soil after the main testing programme (results are contained in Appendix A), thus generating sulphuric acids (this is discussed in more detail in Chapter 4). Another possible mechanism is ion exchange or buffering reactions; occurring within the soil such as the classic lime stabilization reactions, these reactions are detailed by Rogers et al (1997). This paper suggests that in some London Clays upto 5% lime may be required to compensate for the initial consumption of lime within the soil. If this were the case for the London Clay used in this study then the lime added as part of the binder would have been insufficient. The soil structure was also seen to be an influence on the effectiveness of the binder. In the case of the Peat the very open structure of the soil did not promote the formation of bonds between the fibres it was composed of. In the case of the silty sand the course grain size allowed pore spaces for hydrated cement minerals to form with enough surface contact between the grains to allow strong bonds to form. In stiff over consolidated clays, the action of mixing may break up the structure and possible suctions, hence reducing the strength in a way which cannot be recovered. The way in which the mineralogy of the soil and binder interact and the way the soil skeleton affects the strength of the cured soil are discussed in detail in Chapter 4.

Mineralogical analysis conducted upon the soil mix samples was less conclusive than the testing conducted in chapter 4. However, this is understandable as whilst in chapter 4 pure binder samples were analysed in this case the majority of the samples consisted of clay, sand and organic material making the identification of specific hydrated cement minerals much more difficult.

The test programme was successful in terms of proving the potential for Red Gypsum – GGBS binders to improve a range of soils. The strengths achieved were sufficient to pass typical civil engineering specifications for ground improvement in most cases. The slow (when compared to Portland cement) rate of hardening was noted as a potential problem though, and it was identified that for the binder to be used commercially durability of the soils improved with Red Gypsum – GGBS binders should be assessed.

**Concrete Blockwork**

The concrete blockwork testing programme proved that Red Gypsum – GGBS binders could be effective in the production of mass concrete. The strengths achieved by the Red Gypsum – GGBS blocks were lower than the strengths achieved by the Portland cement blocks but the difference was relatively low. It should also be noted that in this programme the longest curing period tested was 28 days. It has been shown in the laboratory soil mixing test programme that Red Gypsum – GGBS sample continue to develop strength up to 56 days, therefore it may be that the long term strength of the Red Gypsum – GGBS blocks would in fact be as high as Portland cement.

More water was required to achieve sufficient workability in the Red Gypsum samples and as is noted in chapter 6 this can be attributed to particle size and shape within the mix. The increased water contents used because of this will have contributed to the lower strength exhibited by the Red Gypsum – GGBS blocks when compared with the Portland cement blocks.

Replacing standard building sand with ICON sand had little impact upon the strength of the cubes and from this point of view there is no reason why ICON sand should not be used in the production of concrete blocks. Ochre also had little effect on the strength of the blocks and showed potential to be used as a colouring agent within paving blocks.

Two issues remained outstanding from the concrete blockwork programme. Firstly, within this research programme it was not possible to conduct mineralogical analysis

on the cured blocks this needs to be done to identify what reactions are occurring whilst the blocks cure. It is predicted that as with the binder samples and the soil mix samples the minerals present within the blocks will be consistent with pozzolanic reactions. The second outstanding issue is one of durability, again within the study it was not possible to conduct freeze-thaw and wet dry durability tests upon the samples. The durability of the samples would need to be verified before Red Gypsum based binder could be used commercially for the production of concrete paving blocks or as a mass concrete.

**Field Trial**

The field trial demonstrated that a Red Gypsum - GGBS binder can be substituted for OPC in the dry mix process without any modifications to existing plant and equipment or a reduction in the efficiency of the installation process. No extra operator training or personal protective equipment is required. This would be important if Red Gypsum binders are to be successful commercially as the cost of developing and distributing new equipment would be prohibitive.

The average 7-day strengths of Red Gypsum - GGBS columns were similar to those achieved in the OPC columns. Longer term strengths were found to be in excess of the OPC columns when the proportion of binder was increased from 200 kg/m$^3$ to 250 kg/m$^3$. The increase in the proportion of the binder reduced the strain to failure, but post peak strengths remained in excess of the design specification. Hence the alternative binder was at least as effective as OPC as a stabilising agent for peaty CLAY.

From the tests performed during this study, the strength of GGBS-red gypsum columns tested did not appear to be significantly detrimentally affected by soaking, freeze-thaw or wet-dry cycles so it may be argued that the columns are reasonably durable. Again, the alternative binder produced columns that were at least as durable as the cement columns under the same conditions. However, only a limited number of samples could be tested and further testing of the long-term strength characteristics is recommended. These results are encouraging from the point of view of the research

as a whole, as they indicate that Red Gypsum – GGBS cemented materials can be durable in freeze-thaw and wet-dry conditions.

Mineralogical investigations have shown that the minerals formed in the samples from the GGBS – red gypsum columns were of the type formed by pozzolanic reactions. It can therefore be reasonably concluded that pozzolanic reactions between the GGBS and the red gypsum were taking place. This was further evidenced by the observed pH dependence of the strength of the GGBS-red gypsum columns. This is again consistent with the previous testing programmes.

pH was again shown to be a critical factor in the effectiveness of the Red Gypsum – GGBS binder. It is recommended that, if there is a possibility for oxidising reactions to reduce the pH of soil samples, these must be stored in air-tight containers prior to initial design mix testing. Furthermore, if there is any doubt over the pH of the soil-binder mixes this should be adjusted (using a small addition of lime) during the treatment process.

**Comparison of Results**

Figure 8.1 shows a summary of the maximum strength results from each of the phases of the testing programme.

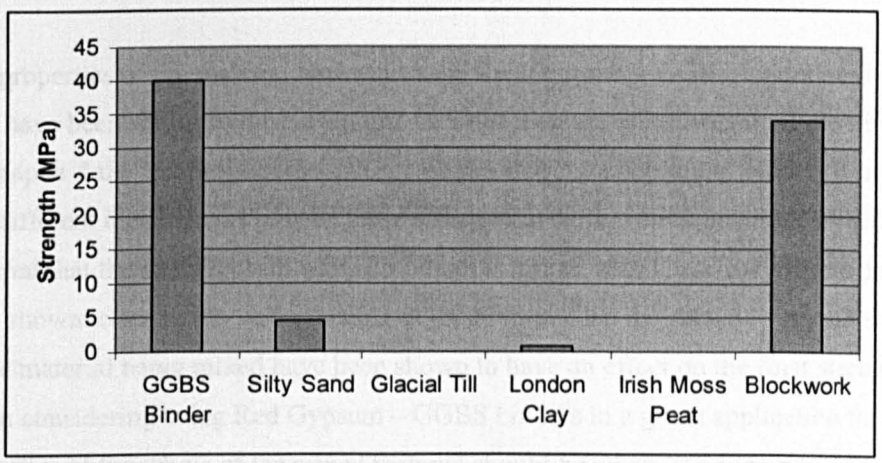



Figure 8.1 Comparison of maximum strength results achieved in each part of the testing programme. (Results for GGBS Binder and Blockwork are Unconfined Compressive Strength, soil results are shear strengths).

The figure shows that when used as pure binder or as a replacement for Portland cement in concretes that the strengths were significantly higher than for the soil mixing (Glacial Till and Irish Moss Peat strengths do not register on the graph). This illustrates that the effectiveness of the binder is significantly dependant upon the material it is mixed with.

## 8.2 General Discussion

By considering the results from all four of the test programmes in this study it is possible to identify that there are several factors that influence the effectiveness of Red Gypsum – GGBS and its potential for use as a construction material. These factors will be discussed in detail in this section but can be summarised as follows.

- Specification for final material properties.
- Properties of the material that the Red Gypsum – GGBS binder is mixed with.
- Conditions that mixing takes place under.
- What is the motivation for using Red Gypsum - GGBS as a construction material.

### 8.2.1 Properties of the material with which Red Gypsum – GGBS binder is mixed

The properties of the material with which the Red Gypsum – GGBS binder are mixed with have been shown to have a significant impact on the effectiveness of the binder. In Chapter 4 the test programme focused on developing an optimum binder. It may be that different binder ratios may be more effective in certain circumstances. When the material that the Red Gypsum – GGBS binder is mixed with has a low pH this has been shown to retard the strengthening of the binder. Also the particle size and shape of the material being mixed have been shown to have an effect on the final strength. When considering using Red Gypsum – GGBS binders in a given application the properties of the whole of the mixed material should be taken into account, particularly the mineralogy and particle size/shape properties.

### 8.2.2 Conditions under which mixing takes place

Difference in material properties between samples mixed in-situ and samples mixed in the laboratory have highlighted the fact that the environment in which the materials are mixed will effect final properties of the cured material. When mixing Red Gypsum – GGBS with Peat it is clear that the confining pressure under which curing takes place significantly effects the properties of the cured soil. It was also noted that during laboratory soil mixing tests, Red Gypsum – GGBS samples cured in soaked conditions achieved lower strengths than samples cured un-soaked. Again this should be taken into account when considering Red Gypsum – GGBS binder for use in construction projects.

### 8.2.3 Specification for final material properties

If Red Gypsum – GGBS binders are to be used they will have to meet civil engineering specifications. These are individual for each construction project. The results from the testing programmes have shown that the Red Gypsum – GGBS binders to not always achieve them same high strengths as Portland cements and do not achieve these strengths as fast. If, however, a specification does not require significantly high strengths or fast hardening times then Red Gypsum – GGBS does not need to out perform Portland cements in order to be useful.

Also, as has been shown in the Blockwork phase of the research programme Portland cement can be added to the Red Gypsum – GGBS binder to increase the initial rate of hardening. Portland cements have been developed over several decades so that now many types are available, sulphate resistant, fast hardening, frost resistant etc. It is possible that with further development Red Gypsum – GGBS binders could be developed in a similar way by adding admixtures so that they can meet a wide variety of engineering specifications.

### 8.2.4 Motivation for using Red Gypsum - GGBS as a construction material

Finally when considering the effectiveness of the Red Gypsum – GGBS binder it is important to note that as well as the engineering considerations there are also

commercial and environmental considerations. In this study Portland cement has been used as a comparison to assess the various properties exhibited by the Red Gypsum – GGBS binder. Whilst there remain questions over the durability of Red Gypsum – GGBS samples in some applications from a strength and stiffness point of view it has been shown that, whilst not always achieving as high a strength in as fast a rate as Portland cement, Red Gypsum – GGBS binder can develop strengths sufficient for typical civil engineering specifications. If strength and stiffness is the only way in which we are measuring the effectiveness of Red Gypsum – GGBS binders then Portland cement is still preferable. Assuming that Red Gypsum – GGBS bound material can be shown to be durable the deciding factors in whether or not it is effective will be:

*Is it environmentally beneficial, in preventing a significant amount of material going to landfill and significant environmental damage that would be caused by the production of a like amount of Portland cement?*

and

*Is it commercially viable when processing, transport and landfill tax costs/benefits are taken into consideration?*

The answer to the first of these questions is quite straightforward. Using Red Gypsum as part of a replacement for Portland cement would have clear environmental benefits. The second question is far more difficult to answer and can only be addressed by conducting commercial scale trials such as the one that Huntsman Tioxide Ltd are doing. Ultimately the success or failure of Red Gypsum as an engineering product will rest on the economy of production costs and landfill tax legislation.

### 8.3 Conclusions and Recommendations

Despite areas highlighted for further study it is felt that a thorough investigation has been made of applications in which Red Gypsum may be used as a construction

material. Publications derived from the study are Hughes and Glendinning (2004) and Hughes and Glendinning (2005) and further publications are planned.

Red Gypsum can be used as a construction material, particularly when mixed with GGBS to form a binder. In order to be a success it will need be commercially competitive when produced on a large scale, the financial viability will depend on the savings made by not disposing to landfill.

The composition of Red Gypsum – GGBS binders will need to be tailored to individual applications, whether this is to take into account a low pH curing environment or the desire for more rapid hardening. Where Red Gypsum – GGBS binders are to be used as an in-situ soil binder then a prior laboratory and site trial would be required.

Whilst this study has shown that Red Gypsum – GGBS binders can be used in the construction industry further research needs to be conducted before they can.

The laboratory testing programme to assess the potential of using Red Gypsum – GGBS binders in concrete blocks was by necessity rather small. In order to test different binders, and types, curing times etc many blocks have to be produced and with each new variable the number of cubes multiplies. Investigating the use of Red Gypsum binders in concrete paving blocks and mass concrete would be an ideal topic for a PhD study in its own right, particularly now the concept has been clearly proved. This would allow a sufficient number of tests to be conducted for all variables to be investigated more fully and for longer curing periods to be assessed.

Durability testing is required on the soil mix samples and on the concrete blocks to show whether or not the material is liable to freeze-thaw or wet-dry damage.

Mineralogical tests were conducted as part of this study but this type of analysis is expensive and therefore only a small number of the total number of samples could be tested(less than 30 out of more than 600). In order to fully understand the reactions taking place within the samples mineralogy should be looked at again using a wider range of analysis tools.

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# Appendix A

# Soil classification property testing data and raw material testing data

# Appendix A Contents



**Sample notation**

GT refers to Glacial Till
LC refers to London Clay
SS refers to Silty Sand
Peat refers to Irish Moss Peat

# ALLIED EXPLORATION & GEOTECHNICS LIMITED

Unit 25 Stella Gill Ind. Est. Pelton Fell, Chester-le-Street, DH2 2RG
Tel No. : 0191 3874700 Fax No. : 0191 3874710


## SAMPLE DESCRIPTION SHEET

| Exploratory Hole No. | Sample Depth | Sample ID | Description | Laboratory Tests/Remarks |
|---|---|---|---|---|
| PH/GT | 0.00 | B1 | Brown slightly sandy slightly gravelly CLAY of intermediate plasticity. | MC PI PSD SED SO3+pH |
| PH/LC | 0.00 | J1 | Grey/brown with some yellow veining sandy CLAY of mudstone. | SO3+pH |
| PH/LC | 0.01 | J2 | Brown sandy CLAY of mudstone. | SO3+pH |
| PH/LC | 0.02 | J3 | Grey/brown with some yellow veining sandy CLAY of mudstone. | SO3+pH |
| PH/LC | 0.03 | J4 | Brown sandy CLAY of mudstone. | SO3+pH |
| PH/LC | 0.04 | B1 | Grey/brown slightly sandy CLAY of intermediate plasticity of mudstone. | MC PI PSD SED SO3+pH |
| PH/SS | 0.00 | B1 | White very silty/clayey SAND/(possibly chalk). (Tested as Clay of low plasticity). | MC PI PSD SED SO3+pH |

Contract Title :- **Nafferton Farm Ground Investigation**

Client :- **University of Newcastle**

Signed :-

Name :-

Page 1 of 1

Date of issue :- 11/08/2005

Certificate No :- SD/3498/1

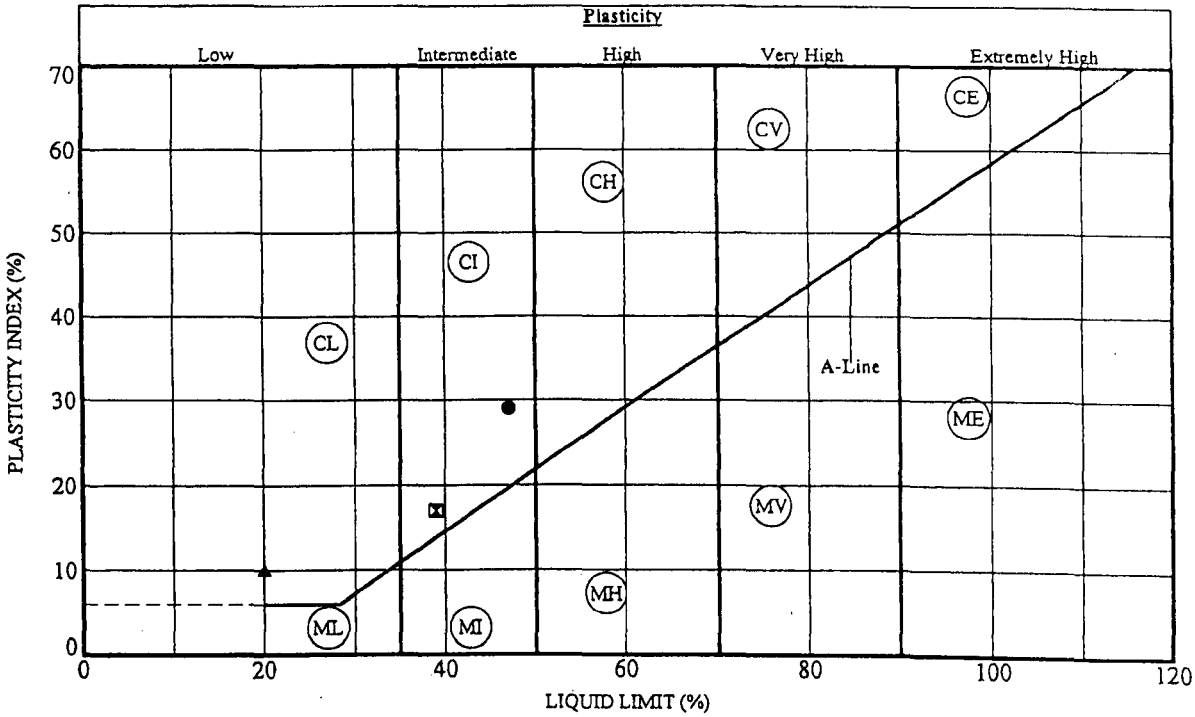
AEG Contract No. :- 3498

UKAS TESTING 1367

ALLIED EXPLORATION & GEOTECHNICS LIMITED
Unit 25 Stella Gill Ind. Est. Pelton Fell, Chester-le-Street, DH2 2RG

# ATTERBERG LIMITS & NATURAL MOISTURE CONTENT

Test Method :- BS 1377 : Part 2 : Clause 3.2, 4.1, 4.3, 4.4 & 5.3 : 1990




| Specimen Identification | | | LL | PL | PI | <0.425mm (%) | m/c (%) | Date Tested | |
|---|---|---|---|---|---|---|---|---|---|
| ● PH/GT | 0.00 | B1 | 47 | 18 | 29 | NAT | 23.9 | 25/07/2005 | |
| ⊠ PH/LC | 0.04 | B1 | 39 | 22 | 17 | 71 | 13 | 25/07/2005 | # |
| ▲ PH/SS | 0.00 | B1 | 20 | 10 | 10 | NAT | 0.5 | 25/07/2005 | |

For description of sample please refer to the Laboratory Sample Description Sheet. # = Insufficient for 4 point PI

| Contract Title :- | Client :- |
|---|---|
| Nafferton Farm Ground Investigation | University of Newcastle |

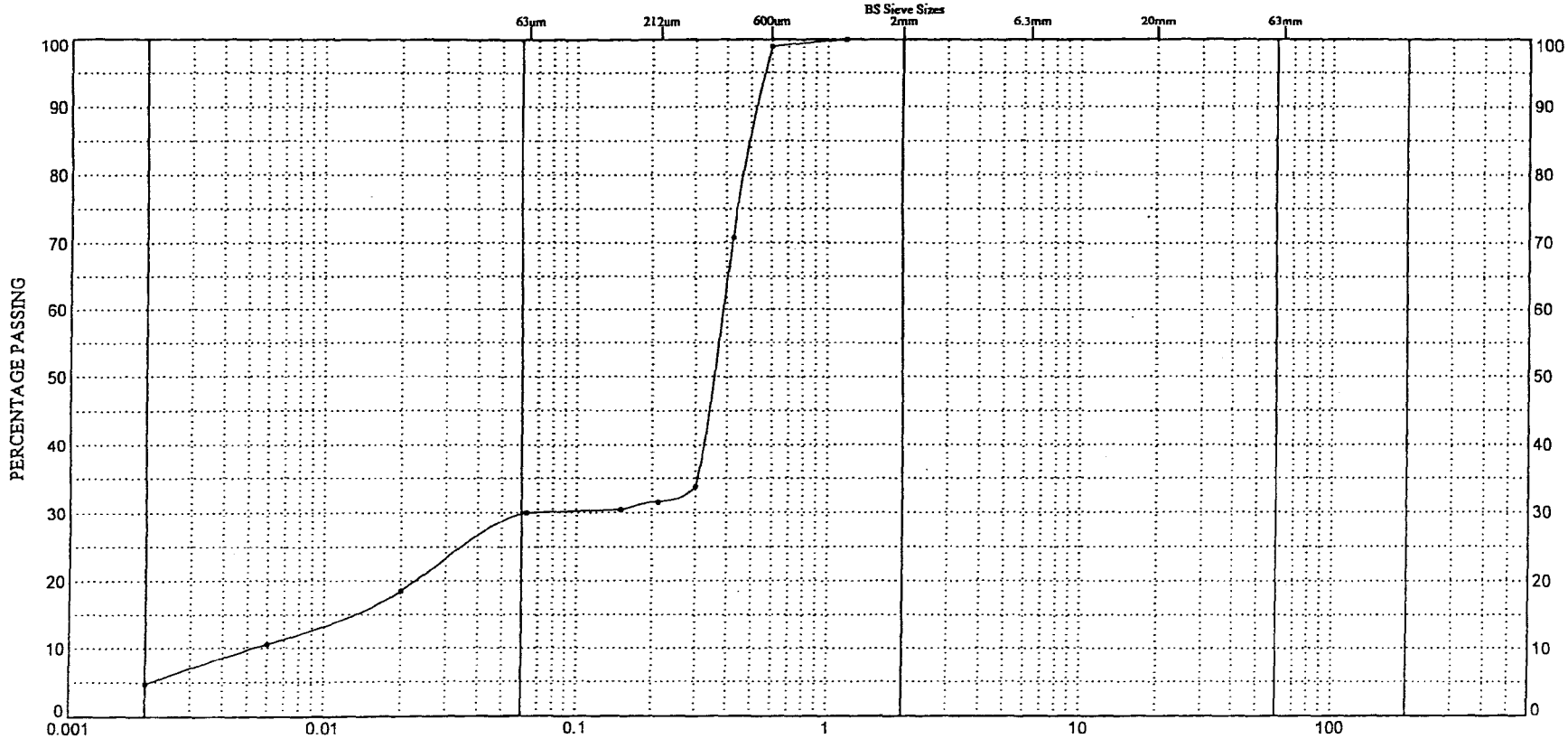
| | | | |
|---|---|---|---|
| Signed :- | Name :- | | Page 1 of 1 |
| Date of issue :- 11/08/2005 | Certificate No :- PI/3498/1 | AEG Contract No :- 3498 | |

UKAS TESTING 1367

ALLEN EXPLORATION & GEOTECHNICS LIMITED
Unit 25 Stella Gill Ind. Est. Pelton Fell, Chester-le-Street, DH2 2RG

| Test Method :- BS1377 : Part 2 : 1990 | Exploratory Hole No :-PH/SS | Depth :- 0.00 | Sample Type & No :- B1 | Date Tested :- 25/07/2005 |
|---|---|---|---|---|

# PARTICLE SIZE DISTRIBUTION CURVE




| CLAY | Fine | Medium | Coarse | Fine | Medium | Coarse | Fine | Medium | Coarse | COBBLES | BOULDERS |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | SILT | | | SAND | | | GRAVEL | | | | |

For description of sample please refer to the Laboratory Sample Description Sheet

| Date of issue :- | Certificate No :- | Signed | Name | Page 1 of 1 |
|---|---|---|---|---|
| 09/08/2005 | PSD/3498/PH/SS/B1/0.00 | | | |

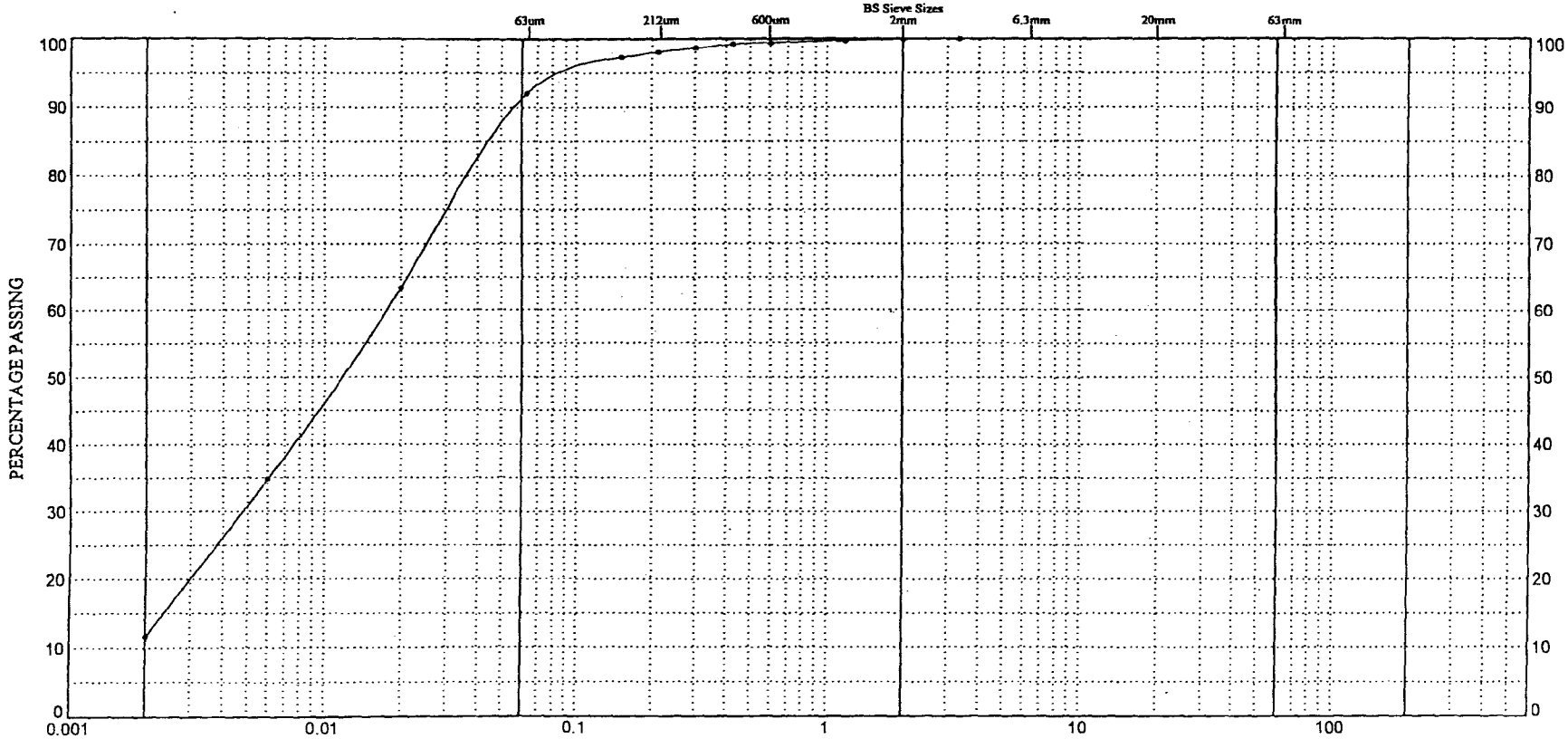
| Client :- | Contract Title :- | AEG Contract No :- |
|---|---|---|
| University of Newcastle | Nafferton Farm Ground Investigation | 3498 |

UKAS TESTING 1367

ALLEN EXPLORATION & GEOTECHNICS LIMITED
Unit 25 Stella Gill Ind. Est. Pelton Fell, Chester-le-Street, DH2 2RG

| Test Method :- BS1377 : Part 2 : 1990 | Exploratory Hole No :-PH/LC | Depth :- 0.04 | Sample Type & No :- B1 | Date Tested :- 25/07/2005 |
|---|---|---|---|---|

# PARTICLE SIZE DISTRIBUTION CURVE




| CLAY | Fine | Medium | Coarse | Fine | Medium | Coarse | Fine | Medium | Coarse | COBBLES | BOULDERS |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | SILT | | | SAND | | | GRAVEL | | | | |

For description of sample please refer to the Laboratory Sample Description Sheet

| Date of issue :- | Certificate No :- | Signed | Name | Page 1 of 1 |
|---|---|---|---|---|
| 09/08/2005 | PSD/3498/PH/LC/B1/0.04 | MSeeno | M. [illegible] | |

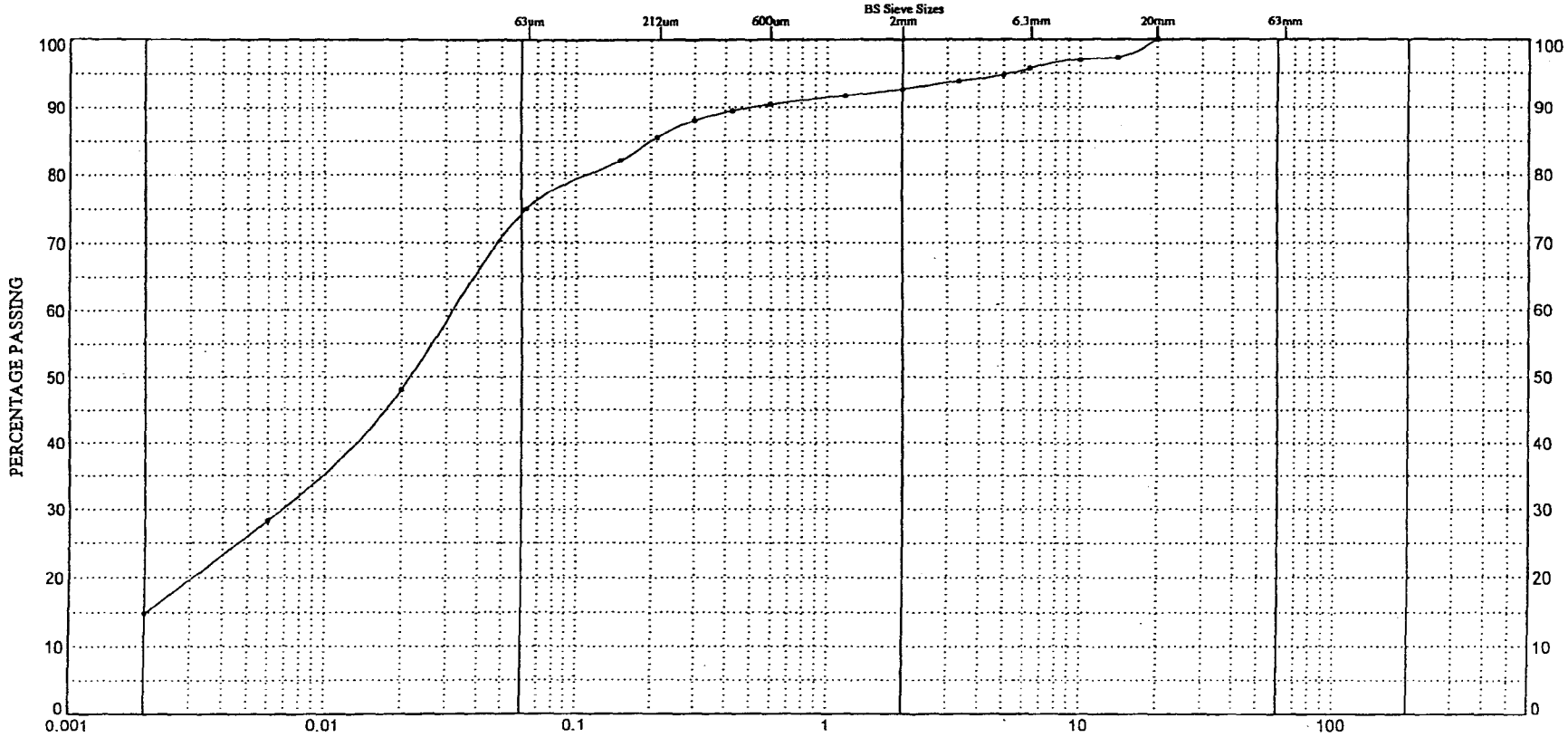
| Client :- | Contract Title :- | AEG Contract No :- |
|---|---|---|
| University of Newcastle | Nafferton Farm Ground Investigation | 3498 |

UKAS TESTING 1367

ALLIED EXPLORATION & GEOTECHNICS LIMITED
Unit 25 Stella Gill Ind. Est. Pelton Fell, Chester-le-Street, DH2 2RG

| Test Method :- BS1377 : Part 2 : 1990 | Exploratory Hole No :-PH/GT | Depth :- 0.00 | Sample Type & No :-B1 | Date Tested :- 25/07/2005 |
|---|---|---|---|---|

# PARTICLE SIZE DISTRIBUTION CURVE




| CLAY | Fine | Medium | Coarse | Fine | Medium | Coarse | Fine | Medium | Coarse | COBBLES | BOULDERS |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | SILT | | | SAND | | | GRAVEL | | | | |

For description of sample please refer to the Laboratory Sample Description Sheet

| Date of issue :- | Certificate No :- | Signed | Name | Page 1 of 1 |
|---|---|---|---|---|
| 09/08/2005 | PSD/3498/PH/GT/B1/0.00 | M Scott | M. SELKIRK | |

| Client :- | Contract Title :- | AEG Contract No :- |
|---|---|---|
| University of Newcastle | Nafferton Farm Ground Investigation | 3498 |

UKAS TESTING 1367

2139

dets
Derwentside Environmental Testing Services

# Certificate of Analysis

**Certificate Number : 05-00958**

| | | | |
|---|---|---|---|
| **Client Reference:** | 3498 | **Date of issue:** | 05/08/2005 |
| **Our Reference:** | 05-00958 | | |
| **Clients Name:** | Allied Exploration & Geotechnics Limited | | |
| **Clients Address:** | Unit 25<br>Stella Gill Industrial Estate<br>Pelton Fell<br>DH2 2RJ | | |

| | |
|---|---|
| **Contract Title:** | Nafferton Farm G.I. |
| **Description:** | 7 soil samples |
| **Date Received:** | 25/07/2005 |
| **Date Commenced:** | 25/07/2005 |
| **Date Completed:** | 05/08/2005 |

**Notes:** Test procedures are identified by prefix DETSn (details available upon request)
Observations and Interpretations are outside the UKAS Accreditation
* Denotes test not included in laboratory scope of accreditation
$ Denotes test carried out by approved subcontractor

**Approved By:**

Richard Bennett — Director

Rob Brown — Laboratory Manager





Derwentside Environmental Testing Services Limited.
Unit 2a/2b, Park Road Industrial Estate South, Consett, Co Durham, DH8 5PY
Tel: 01207 582333 • Fax: 01207 582444 • Email: info@dets.co.uk • www.dets.co.uk
Registered in England No. 370 5643 • VAT No. 708 978 678

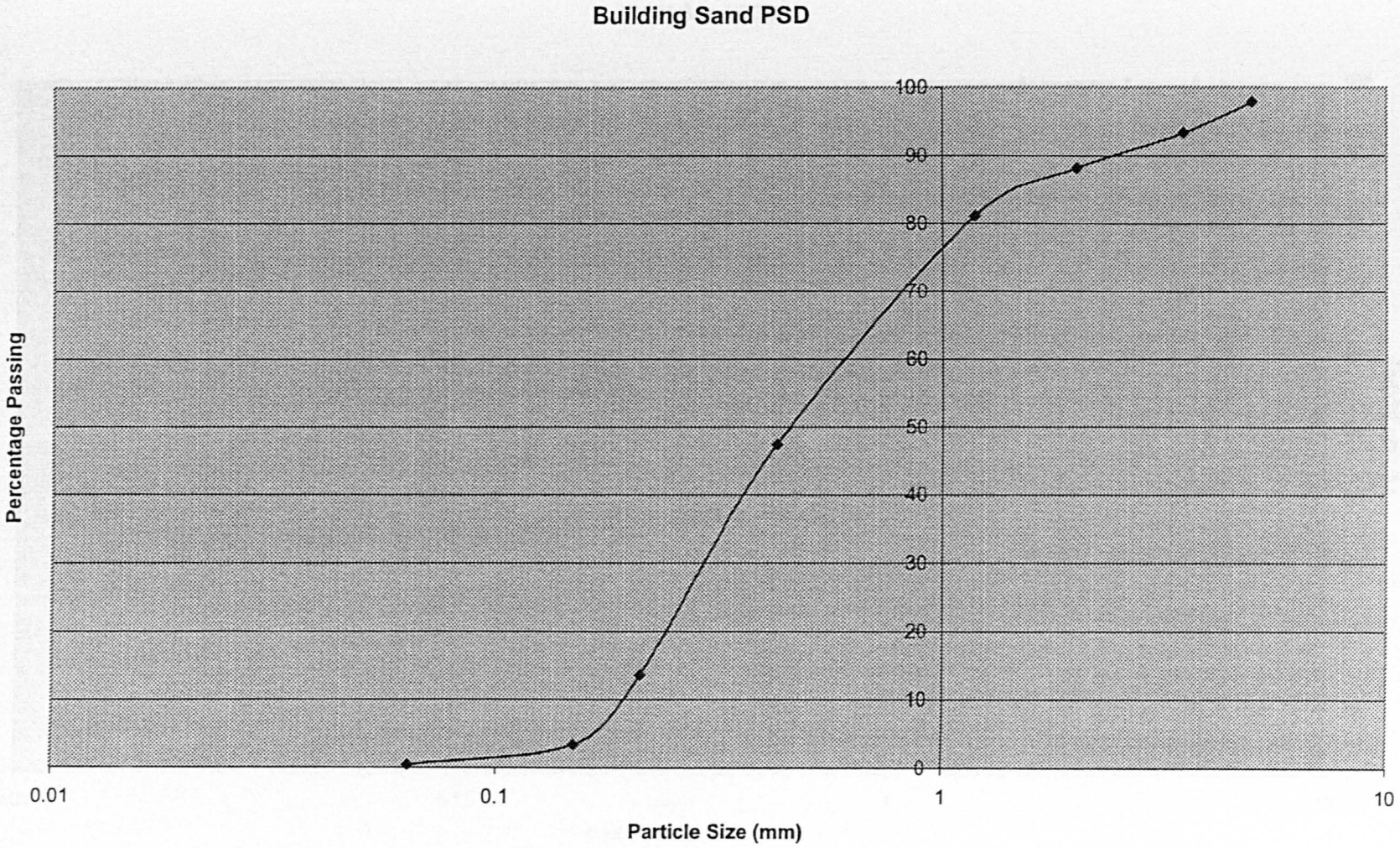
Client Reference 3498

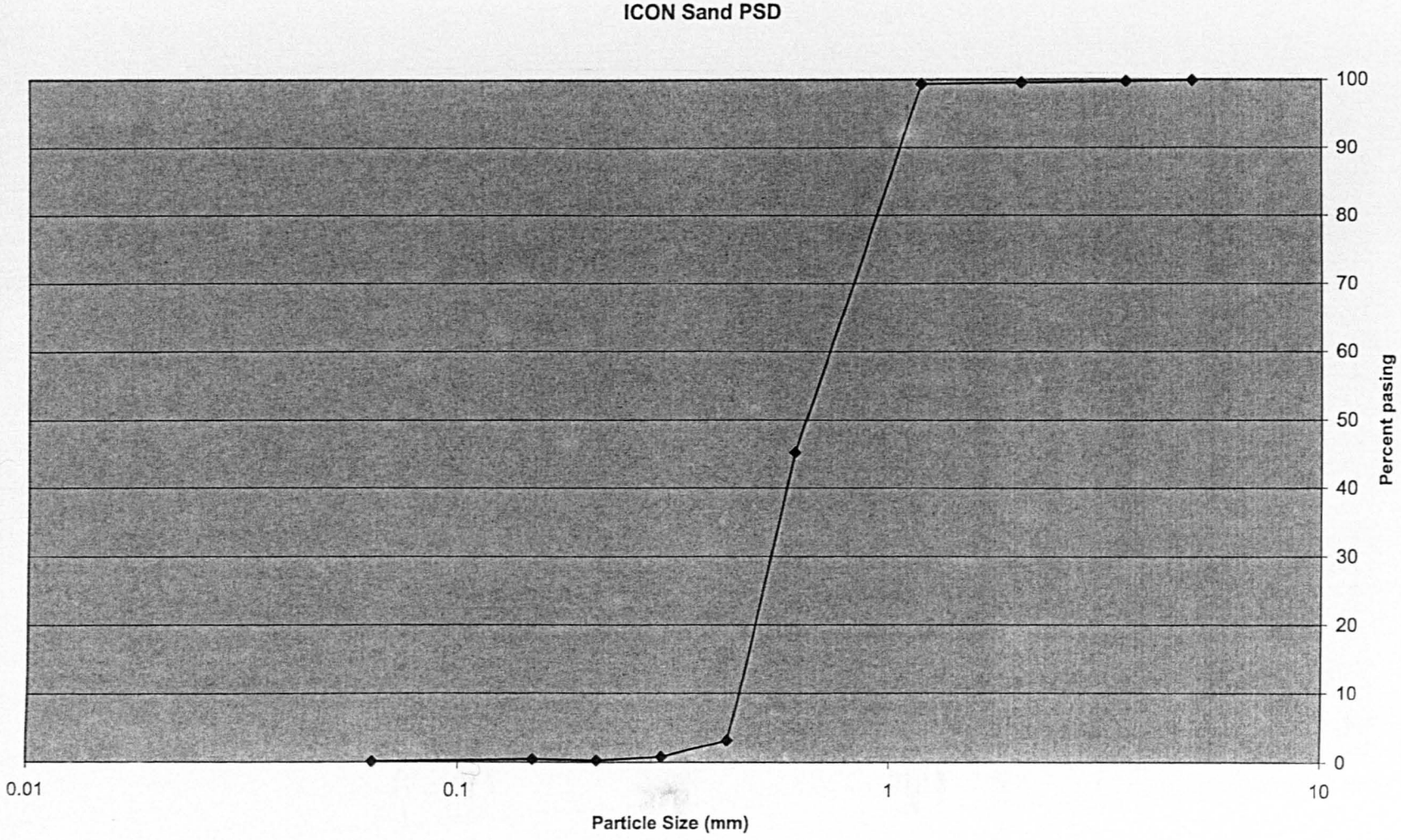
Laboratory Reference 05-00958

## Nafferton Farm G.I.

# Summary of Chemical Analysis

| Borehole or Trial Pit | Depth | Sample No | Sample Type | Lab Ref No | DETS 004 Sulphate Aqueous Extract g/l as SO3 | DETS 004 Sulphate Aqueous Extract g/l as SO4 | DETS 008 pH |
|---|---|---|---|---|---|---|---|
| PH/GT | 0.00 | B1 | Soil | 7029 | 0.65 | 0.78 | 7.9 |
| PH/LC | 0.00 | J1 | Soil | 7030 | 4.40 | 5.29 | 4.8 |
| PH/LC | 0.01 | J2 | Soil | 7031 | 4.83 | 5.79 | 5.2 |
| PH/LC | 0.02 | J3 | Soil | 7032 | 5.18 | 6.22 | 3.4 |
| PH/LC | 0.03 | J4 | Soil | 7033 | 4.55 | 5.46 | 5.7 |
| PH/LC | 0.04 | B1 | Soil | 7034 | 3.49 | 4.19 | 6.7 |
| PH/SS | 0.00 | B1 | Soil | 7035 | 0.23 | 0.28 | 5.8 |


Building Sand PSD
Percentage Passing
100
90
80
70
60
50
40
30
20
10
0
0.01
0.1
1
10
Particle Size (mm)


ICON Sand PSD
100
90
80
70
60
50
40
30
20
10
0
Percent pasing
0.01
0.1
1
10
Particle Size (mm)

# Appendix B

# Binder testing data

# Appendix B Contents

Red Gypsum – PFA binder results.

| RG:PFA ratio | Pre cure Water Content (%) | Unconfined Compressive Strength (MPa) | Post cure water content (%) | Bulk Density ($MgM^3$) | Post Curing Dry Density ($MgM^3$) | Young's Modulus (MPa) | Water Content Loss (%) |
|---|---|---|---|---|---|---|---|
| 50:50 | 9.4 | 0.06 | 7.0 | 1.37 | 1.28 | 10.8 | 24.9 |
| 50:50 | 19.8 | 0.05 | 16.9 | 1.48 | 1.27 | 2.9 | 14.6 |
| 50:50 | 30.23 | 0.009 | 24.1 | 1.88 | 1.52 | 0.31 | 20.3 |
| 50:50 | 40.6 | - | 37.5 | 1.73 | 1.26 | - | - |
| 30:70 | 35.7 | 0.16 | 21.7 | 1.71 | 1.41 | 32.7 | 39.2 |
| 30:70 | 40.5 | 0.34 | 32.4 | 1.64 | 1.24 | 14.2 | 20.1 |
| 30:70 | 53.6 | 0.01 | 35.4 | 1.54 | 1.14 | 2.7 | 34.0 |
| 30:70 | 66.9 | 0.02 | 44.9 | 1.5 | 1.03 | 7.7 | 32.0 |

Red Gypsum – Steel Slag density results.

| RG : Steel Slag ratio | Max Bulk Density (Mg/m3) | @ Water Content (%) | Max Dry Density (Mg/m3) | @ Water Content (%) |
|---|---|---|---|---|
| 90-10 | 1.73 | 40.9 | 1.36 | 30.9 |
| 70-30 | 1.80 | 44.3 | 1.71 | 33.1 |
| 50-50 | 2.05 | 29.8 | 1.71 | 29.8 |
| 30-70 | 2.09 | 18.6 | 1.88 | 18.6 |
| 10-90 | 2.01 | 32.2 | 1.82* | 9.2 |

*no peak observed

Red Gypsum – Steel Slag strength and stiffness results

| RG : Steel Slag ratio | Max UCS (MPa) | @ Water Content (%) | Max Young's Modulus (MPa) | @ Water Content (%) |
|---|---|---|---|---|
| 90-10 | *1.06 | 10.4 | 49.1 | 30.9 |
| 70-30 | *3.41 | 12.2 | 234.3 | 23.2 |
| 50-50 | *3.51 | 21.4 | *274.3 | 21.4 |
| 30-70 | *4.51 | 18.6 | 158.7 | 28.1 |
| 10-90 | *4.23 | 9.2 | *681.1 | 9.2 |

*no peak value observed

Summary of Red Gypsum – Steel Slag binder results

| RG:Steel slag ratio | Pre cure Water Content (%) | UCS (MPa) | Post cure Water Content (%) | Bulk Density (Mg/m3) | Post cure Dry Density (Mg/m3) | Young's Modulus (MPa) | Water Content Reduction .(%) |
|---|---|---|---|---|---|---|---|
| 90:10 | 10.4 | 1.06 | 8.1 | 1.41 | 1.30 | 43.3 | 22 |
| 90:10 | 20.4 | 0.88 | 18.1 | 1.54 | 1.30 | 37.5 | 11.3 |
| 90:10 | 30.9 | 0.61 | 25.9 | 1.71 | 1.36 | 49.1 | 16.3 |
| 90:10 | 40.9 | 0.47 | 36.1 | 1.73 | 1.27 | 12.3 | 11.6 |
| 90:10 | 52.9 | 0.05 | 46.8 | 1.69 | 1.15 | 4.9 | 11.4 |
| 90:10 | 70.4 | 0.01 | 58.1 | 1.62 | 1.02 | 3.7 | 17.5 |
| 70:30 | 12.2 | 3.41 | 7 | 1.61 | 1.5 | 149.3 | 42.7 |
| 70:30 | 23.2 | 2.93 | 14 | 1.74 | 1.68 | 234.3 | 39.8 |
| 70:30 | 33.1 | - | 18.5 | 1.74 | 1.71 | - | 44.1 |
| 70:30 | 44.3 | 0.75 | 30.9 | 1.8 | 1.43 | 120.8 | 30.2 |
| 70:30 | 58.7 | 0.17 | 42.4 | 1.7 | 1.15 | 53.8 | 27.7 |
| 50:50 | 10.4 | - | 5.25 | 1.81 | 1.72 | - | 49.7 |
| 50:50 | 21.4 | 3.51 | 14.1 | 1.93 | 1.68 | 274.3 | 33.8 |
| 50:50 | 29.8 | 3.06 | 20 | 2.05 | 1.71 | 128.1 | 33.1 |
| 50:50 | 42.8 | 0.8 | 29.6 | 1.87 | 1.44 | 102.5 | 31.0 |
| 50:50 | 49.5 | 0.06 | 44.5 | 1.66 | 1.15 | 23.1 | 10.2 |
| 30:70 | 8.4 | - | 4.4 | 1.51 | 1.45 | - | 47.9 |
| 30:70 | 18.6 | 4.51 | 11.2 | 2.09 | 1.88 | 119.2 | 39.6 |
| 30:70 | 28.1 | 1.95 | 19.7 | 1.96 | 1.64 | 158.7 | 29.9 |
| 30:70 | 42.7 | - | 29.4 | 1.85 | 1.43 | - | 31.2 |
| 30:70 | 55.2 | 0.06 | 42.9 | 1.73 | 1.21 | 23.1 | 22.4 |
| 10:90 | 9.2 | 4.23 | 4.6 | 1.91 | 1.82 | 681.1 | 50.3 |
| 10:90 | 18.4 | 2.51 | 13.6 | 1.97 | 1.74 | 129.1 | 26.5 |
| 10:90 | 32.2 | 0.11 | 21.0 | 2.01 | 1.66 | 10.4 | 34.7 |
| 10:90 | 37.3 | 0.09 | 31.0 | 1.88 | 1.44 | 14.7 | 16.8 |

Red Gypsum filter cake – GGBS binder results.

| Red Gypsum : GGBS ratio | Density | | | | UCS | |
|---|---|---|---|---|---|---|
| | Max Bulk Density (Mg/m3) | Water Content (%) | Max Dry Density (Mg/m3) | Water Content (%) | UCS (MPa) | Water Content (%) |
| 90-10 | *1.69 | 41 | - | - | - | - |
| 80-20 | *1.77 | 37.8 | - | - | - | - |
| 70-30 | *1.78 | 32.4 | *1.5 | 40.4 | *16.5 | 40.4 |
| 60-40 | 1.91 | 35.4 | 1.53 | 35.4 | *22.8 | 35.4 |
| 50-50 | *1.89 | 22.8 | *1.61 | 22.8 | 28.63 | 39.6 |
| 40-60 | 1.85 | 33.6 | 1.57 | 24.6 | 15.52 | 24.6 |
| 30-70 | 1.92 | 23.3 | 1.61 | 31.5 | 24.1 | 31.5 |
| 20-80 | 1.89 | 26.9 | 1.51 | 26.9 | 25.6 | 53.9 |
| 10-90 | 2.00 | 22.8 | 1.70 | 22.8 | 39.7 | 35.4 |

*no peak observed

Red Gypsum filter cake – GGBS, summary of results

| RG:GGBS ratio | Water Content (%) | Unconfined Compressive Strength (MPa) | Post Curing Water Content (%) | Bulk Density (MgM3) | Post Curing Dry Density (MgM3) |
|---|---|---|---|---|---|
| 90:10 | 41 | Failed in prep | 33.5 | 1.69 | - |
| 90:10 | 48.4 | Failed in prep | 39.7 | 1.62 | - |
| 90:10 | 56.3 | Failed in prep | 48.6 | 1.55 | - |
| 80:20 | 37.8 | Failed in prep | - | 1.77 | - |
| 80:20 | 44.9 | Failed in prep | - | 1.68 | - |
| 80:20 | 52.2 | Failed in prep | 37.18 | 1.62 | - |
| 80:20 | 59.4 | Failed in prep | 47.84 | 1.59 | - |
| 70:30 | 32.4 | 13.28 | 16.24 | 1.78 | 1.42 |
| 70:30 | 40.4 | 16.47 | 17.83 | 1.76 | 1.5 |
| 60:40 | 29.4 | Failed in prep | 9.6 | 1.88 | - |
| 60:40 | 35.4 | 22.8 | 16.6 | 1.91 | 1.53 |
| 60:40 | 53.0 | 15.49 | 25.34 | 1.58 | 1.34 |
| 60:40 | 60.1 | 10.4 | 23.9 | 1.55 | 1.27 |
| 50:50 | 22.8 | 12.38 | 10.14 | 1.89 | 1.61 |
| 50:50 | 30.9 | 20.39 | 14.95 | 1.86 | 1.58 |
| 50:50 | 39.6 | 28.63 | 16.06 | 1.76 | 1.49 |
| 50:50 | 51.3 | 22.55 | 23.05 | 1.7 | 1.44 |
| 50:50 | 83.1 | 13.52 | 29.37 | 1.57 | 1.33 |
| 50:50 | 94.0 | 11.24 | 40.06 | 1.54 | 1.3 |
| 40:60 | 18.2 | 9.84 | 10.87 | 1.76 | 1.51 |
| 40:60 | 24.6 | 15.52 | 15.9 | 1.83 | 1.57 |
| 40:60 | 33.6 | 15.06 | 19.7 | 1.85 | 1.56 |
| 40:60 | 42.4 | 9.92 | 25.11 | 1.66 | 1.4 |
| 30:70 | 11.0 | 15.87 | 5.17 | 1.84 | 1.34 |
| 30:70 | 23.3 | Failed in prep | 10.33 | 1.92 | - |
| 30:70 | 31.5 | 24.08 | 15.43 | 1.88 | 1.61 |
| 30:70 | 39.5 | 19.81 | 17.23 | 1.77 | 1.51 |
| 30:70 | 47.6 | 10.83 | 25.55 | 1.73 | 1.47 |
| 20:80 | 9.5 | Failed in prep | - | 1.56 | - |
| 20:80 | 17.7 | Failed in prep | - | 1.75 | - |
| 20:80 | 26.9 | 19.65 | 12.83 | 1.89 | 1.51 |
| 20:80 | 38.6 | Failed in prep | - | 1.83 | - |
| 20:80 | 53.9 | 25.55 | 21.36 | 1.72 | 1.3 |
| 20:80 | 71 | 16.69 | 34.62 | 1.62 | 1.3 |
| 10:90 | 4.7 | Failed in prep | - | 1.52 | - |
| 10:90 | 12.9 | 10.26 | 6.26 | 1.81 | 1.46 |
| 10:90 | 22.8 | 33.46 | 11.72 | 2.00 | 1.7 |
| 10:90 | 35.4 | 39.68 | 17.76 | 1.92 | 1.62 |
| 10:90 | 50.3 | 23.19 | 27.98 | 1.74 | 1.48 |
| 10:90 | 64.4 | 17.01 | 38.38 | 1.67 | 1.42 |

| RG:GGBS ratio | Water Content (%) | Unconfined Compressive Strength (MPa) | Post Curing Water Content (%) | Bulk Density (MgM3) | Post Curing Dry Density (MgM3) |
|---|---|---|---|---|---|
| 70:30 | 47.5 | 4.92 | 25.6 | 1.68 | 1.46 |
| 70:30 | 47.5 | Failed in Prep | 26.5 | 1.65 | 1.43 |
| 60:40 | 40.7 | 6.12 | 32.1 | 1.64 | 1.42 |
| 60:40 | 40.7 | 7.72 | 30.9 | 1.65 | 1.40 |
| 50:50 | 38.3 | 10.9 | 22.9 | 1.74 | 1.53 |
| 50:50 | 38.3 | 10.8 | 23.1 | 1.73 | 1.50 |
| 40:60 | 48.0 | 11.02 | 26.7 | 1.66 | 1.48 |
| 40:60 | 48.0 | 8.87 | 28.5 | 1.69 | 1.46 |
| 30:70 | 42.5 | 11.83 | 23.3 | 1.73 | 1.41 |
| 30:70 | 42.5 | 18.08 | 24.6 | 1.75 | 1.50 |
| 20:80 | 43.1 | 15.79 | 24.4 | 1.77 | 1.50 |
| 20:80 | 43.1 | 8.99 | 24.9 | 1.78 | 1.50 |
| 10:90 | 41.5 | 23.26 | 25.7 | 1.82 | 1.10 |

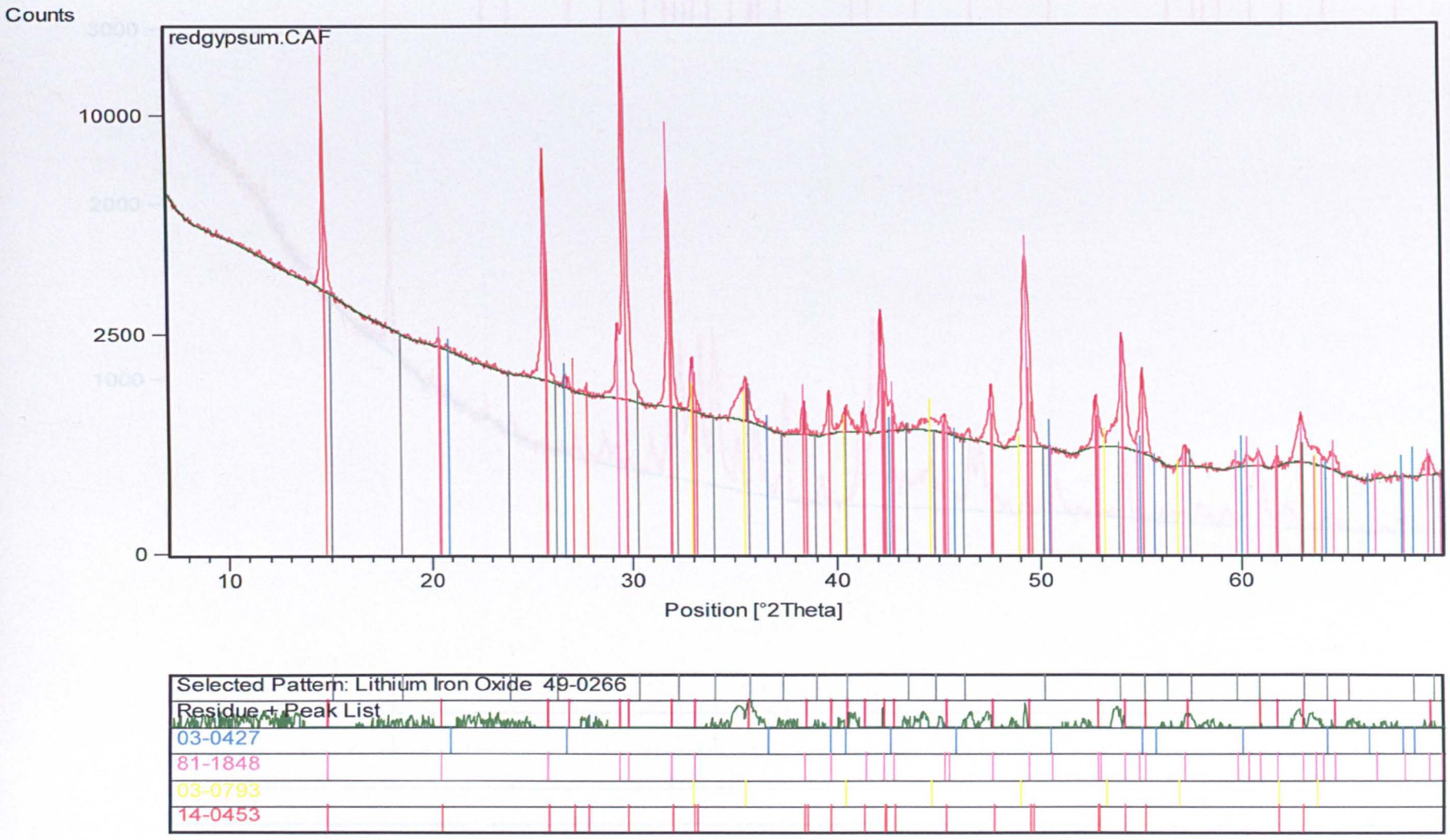
Table 4.9, 2nd Phase Red Gypsum – GGBS binder full results.

**XRD Data**

On plots where the patterns at the base of the plot are not labelled, these codes indicate which mineral is present.

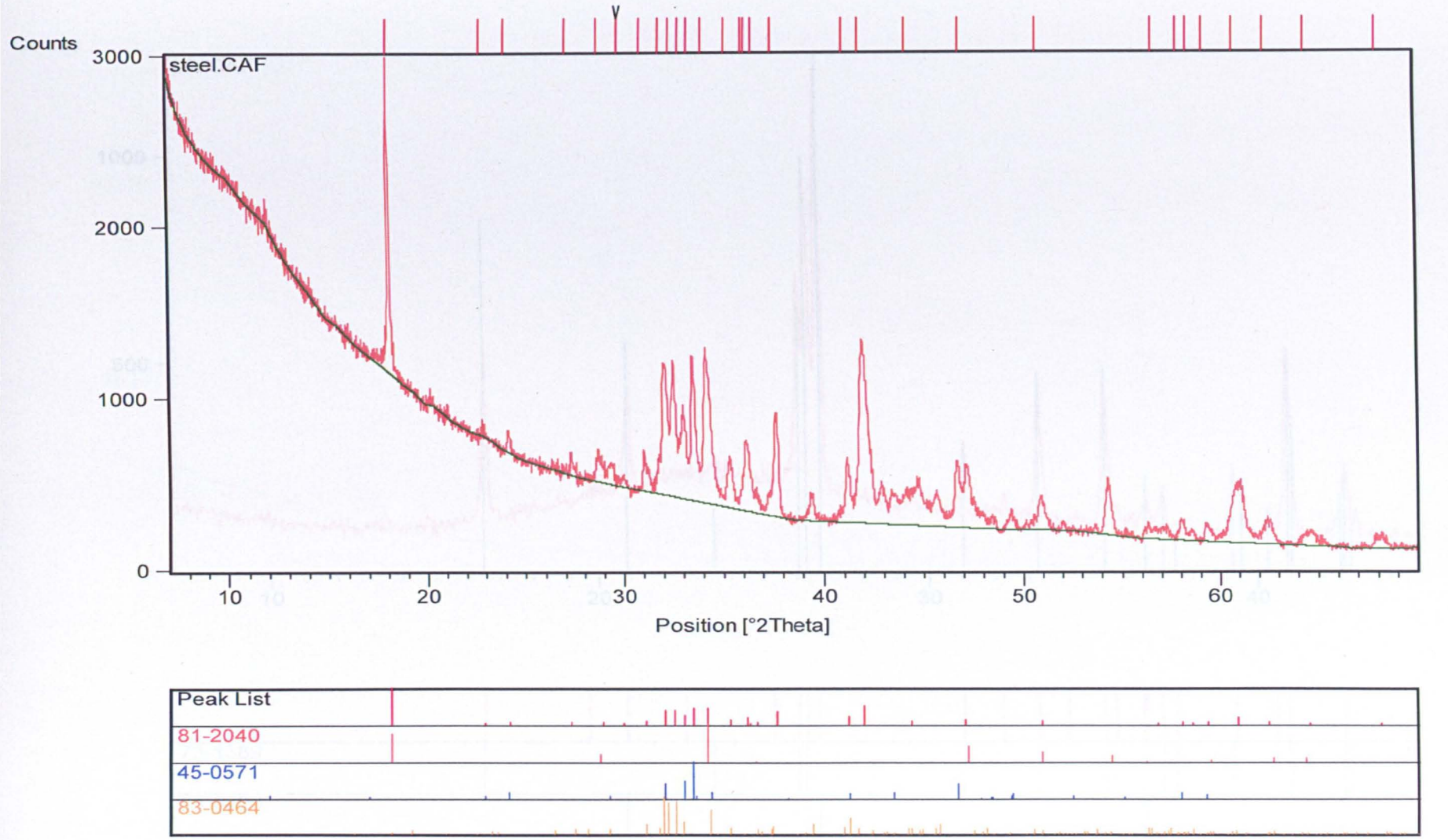
05-0490 Quartz Low
45-0571 Calcium magnesium
70-0388 Calcium silicate
70-2438 Thaumasite
72-0156 Portlandite, synthetic
72-0646 Etteringite
72-1650 Calcite
73-1389 Mullite
73-1765 Rutile, synthetic
74-1433 Gypsum
74-1877 Lepidocrocite
75-1677 Gehlenite, synthetic
76-0841 Akermanite
79-1910 Quartz
81-2040 Calcium Hydroxide
83-0464 Calcium silicate

Red Gypsum


Counts
redgypsum.CAF
10000
2500
0
10
20
30
40
50
60
Position [°2Theta]
Selected Pattern: Lithium Iron Oxide 49-0266
Residue+Peak List
03-0427
81-1848
03-0793
14-0453

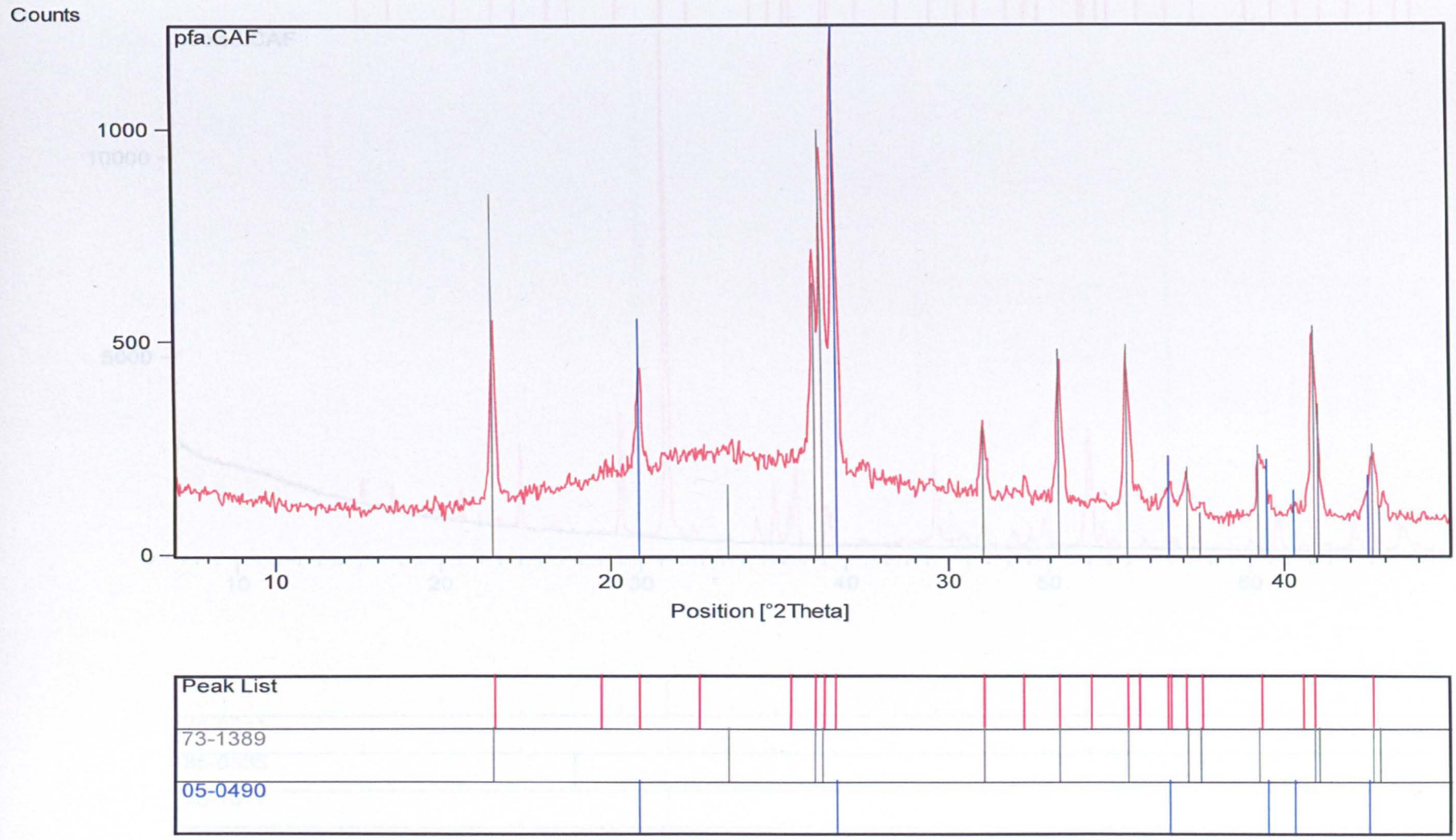
steel slag

Date: 15/11/2005 Time: 16:38:37 File: steel.CAF User: npnh1



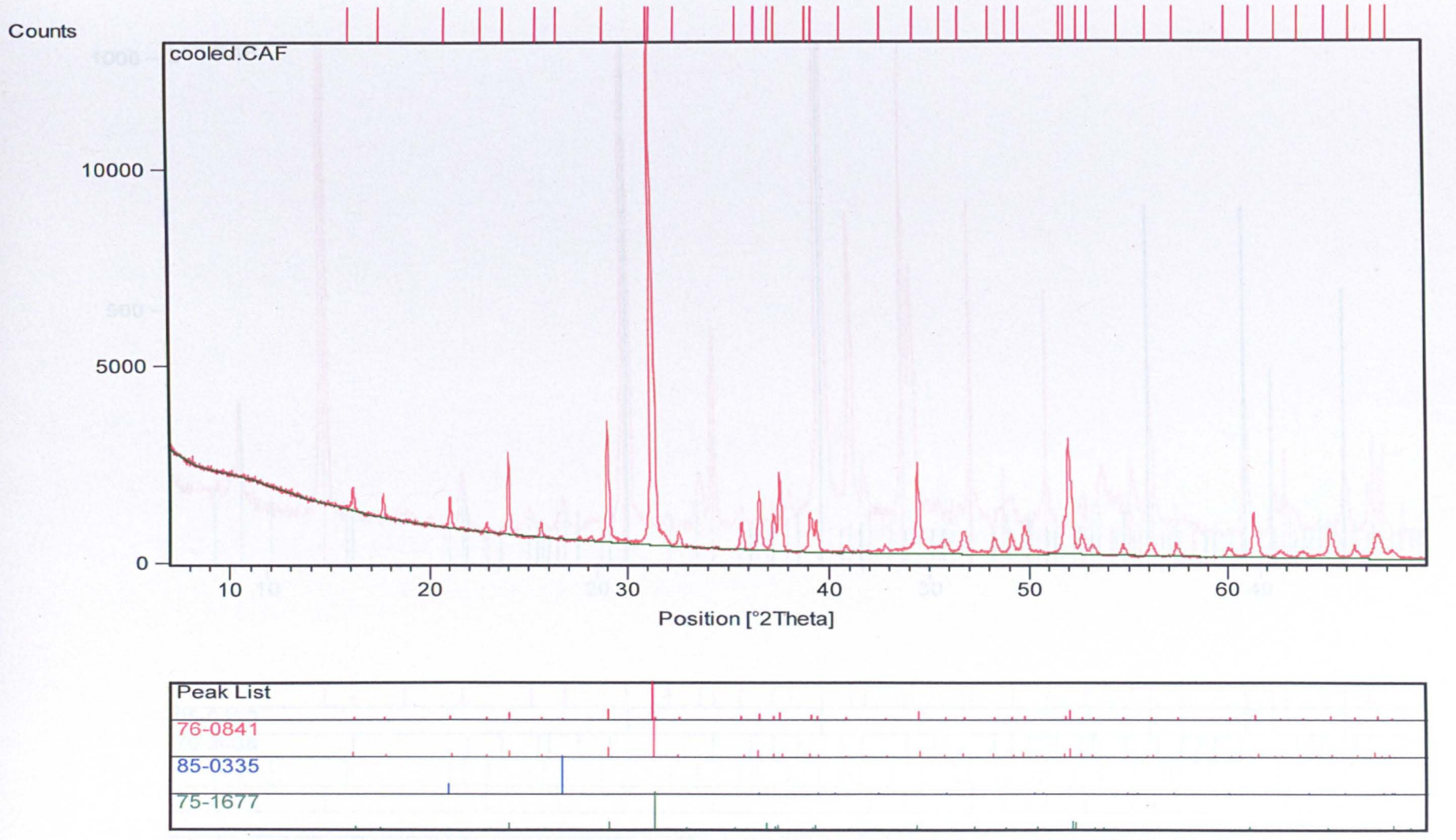
Pulverised Fuel ash

Date: 15/11/2005 Time: 16:37:55 File: pfa.CAF User: npnh1



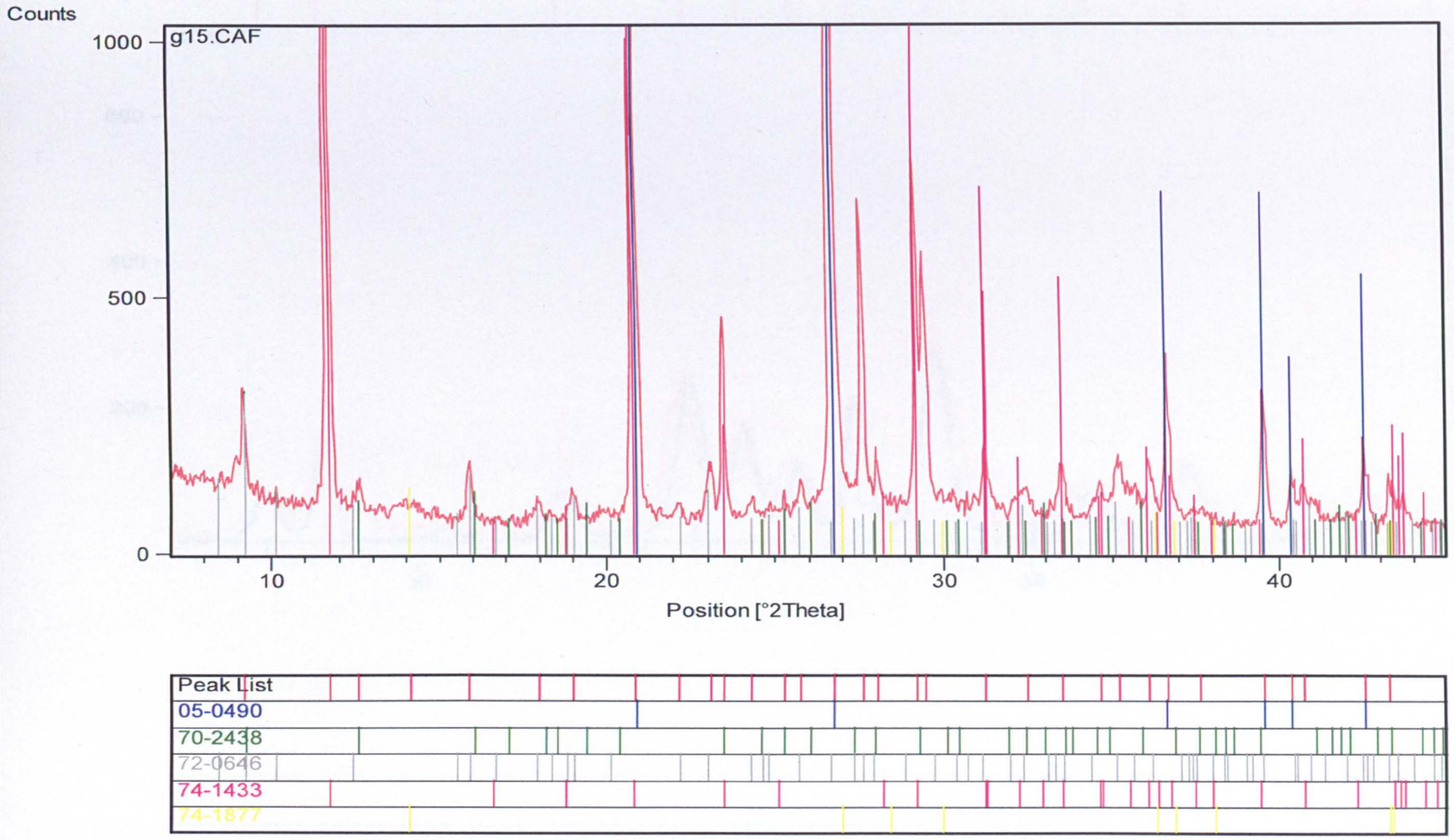
Air cooled blastfurnace slag

Date: 15/11/2005 Time: 16:36:47 File: cooled.CAF User: npnh1



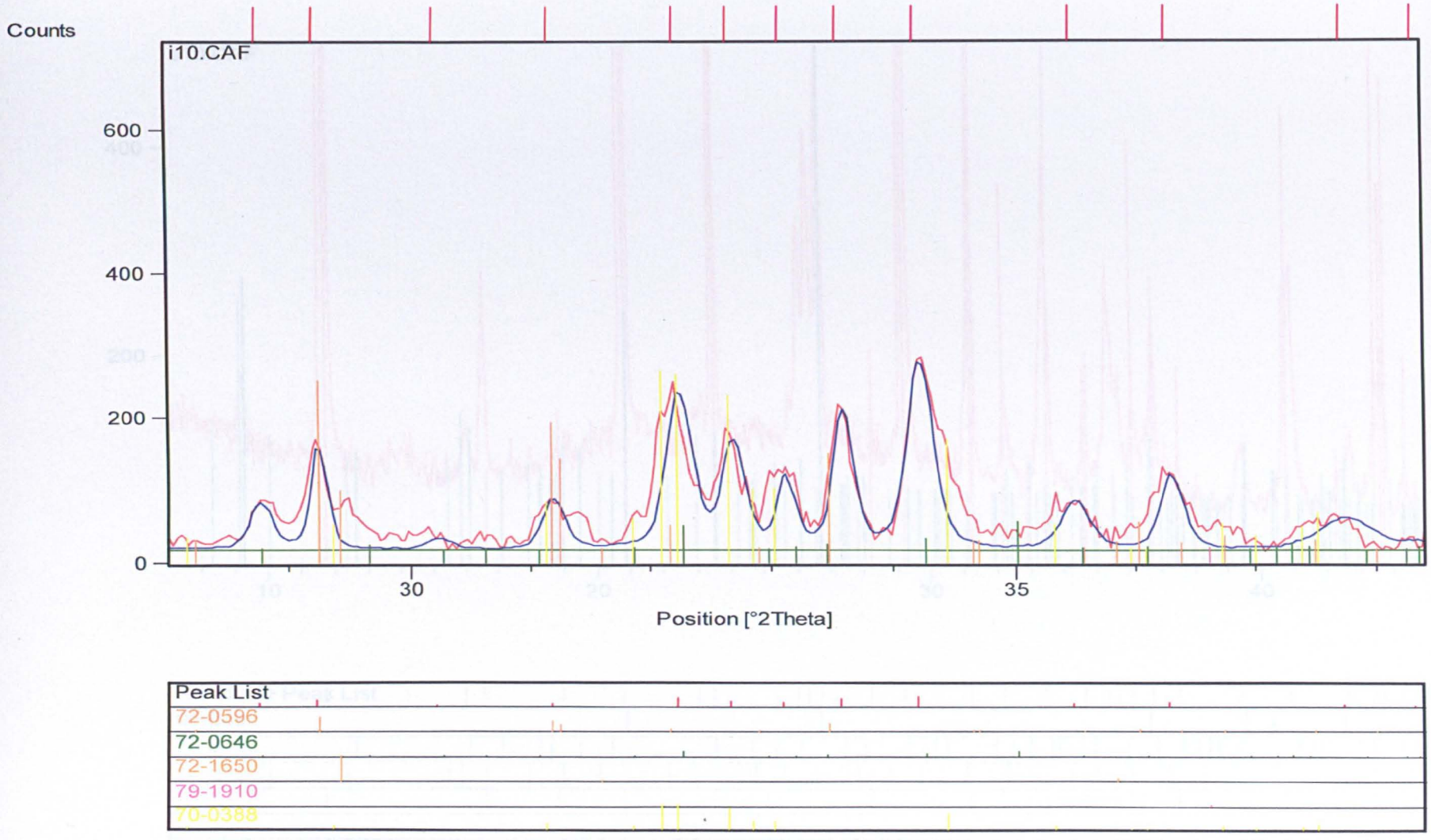
30% RG 70% steel slg

Date: 15/11/2005 Time: 16:41:43 File: g15.CAF User: npnh1

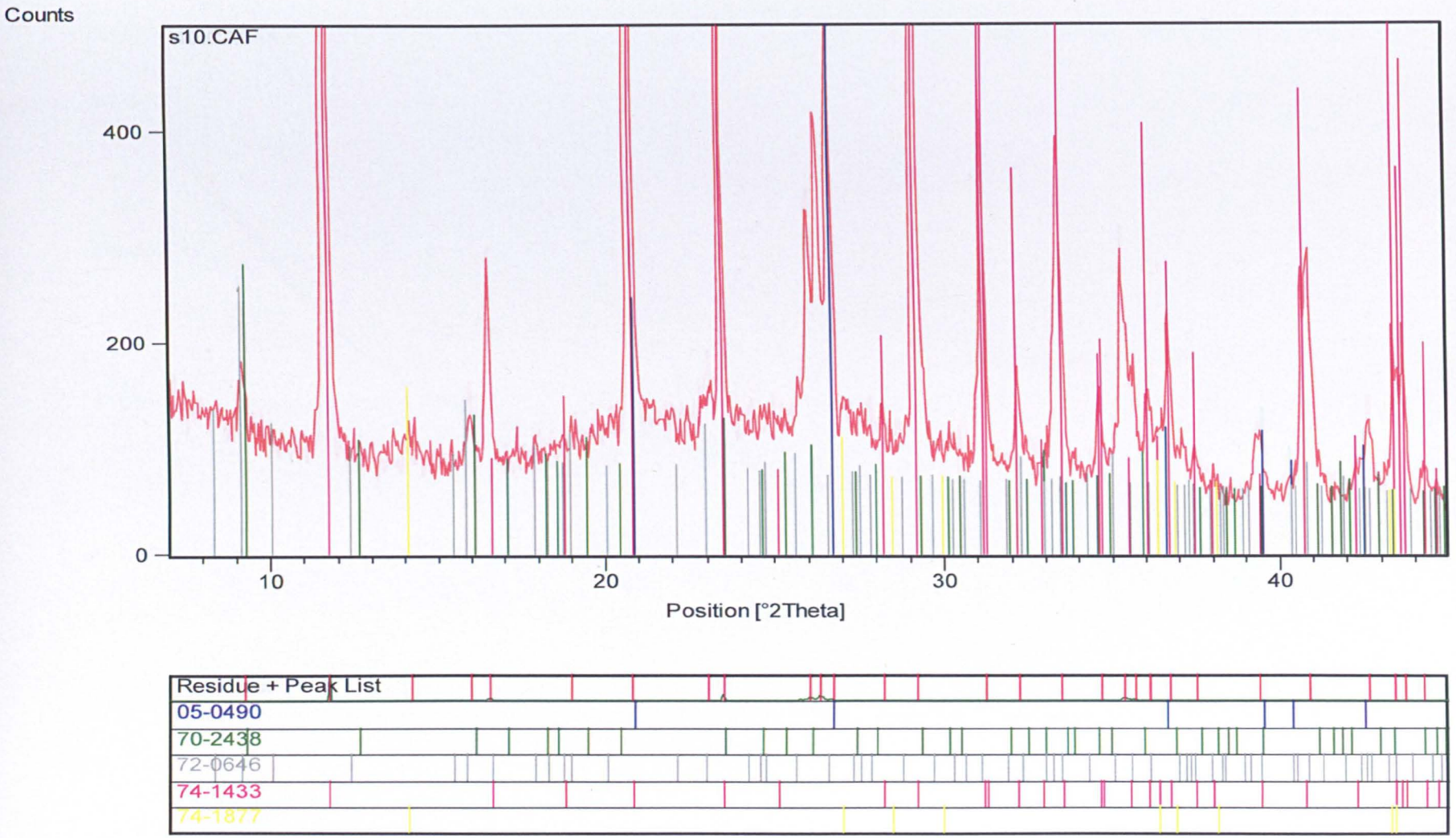


10% RG - 90% steel slag

Date: 15/11/2005 Time: 16:42:12 File: i10.CAF User: npnh1

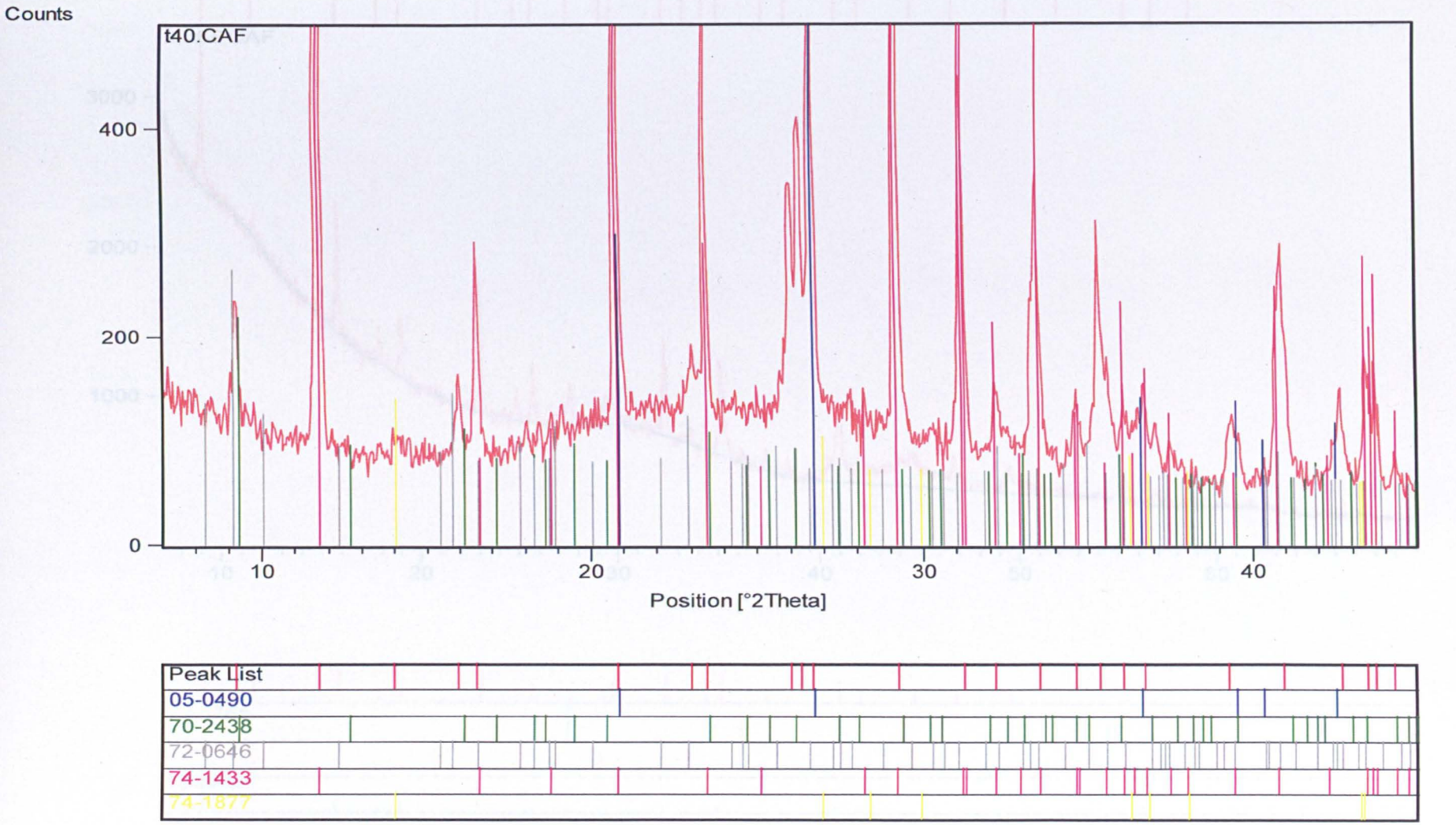


50% RG - 50% PFA


Counts
s10.CAF
400
200
0
10
20
30
40
Position [°2Theta]
Residue + Peak List
05-0490
70-2438
72-0646
74-1433
74-1877

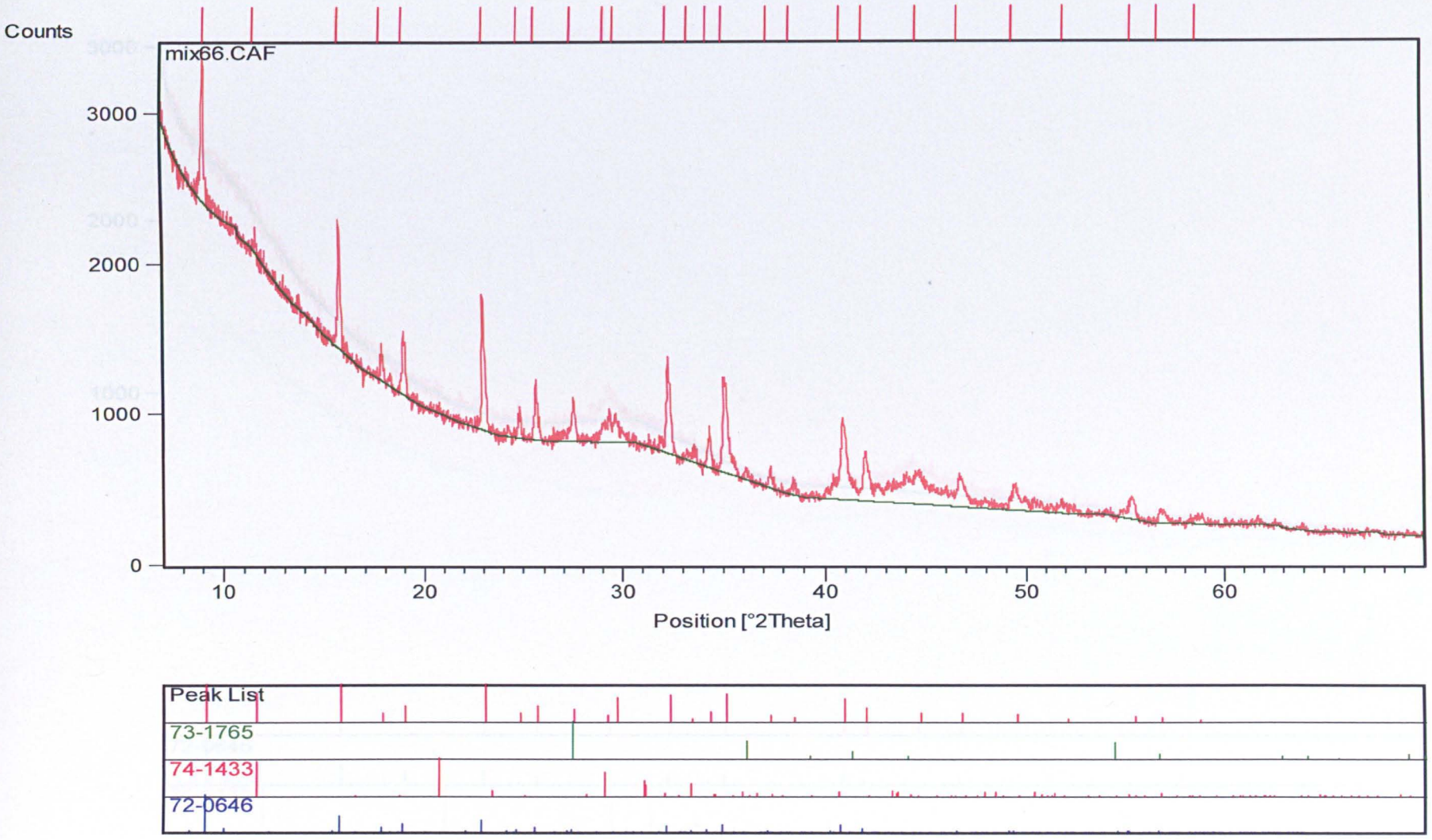
30% RG - 70% PFA

Date: 15/11/2005 Time: 16:43:15 File: t40.CAF User: npnh1



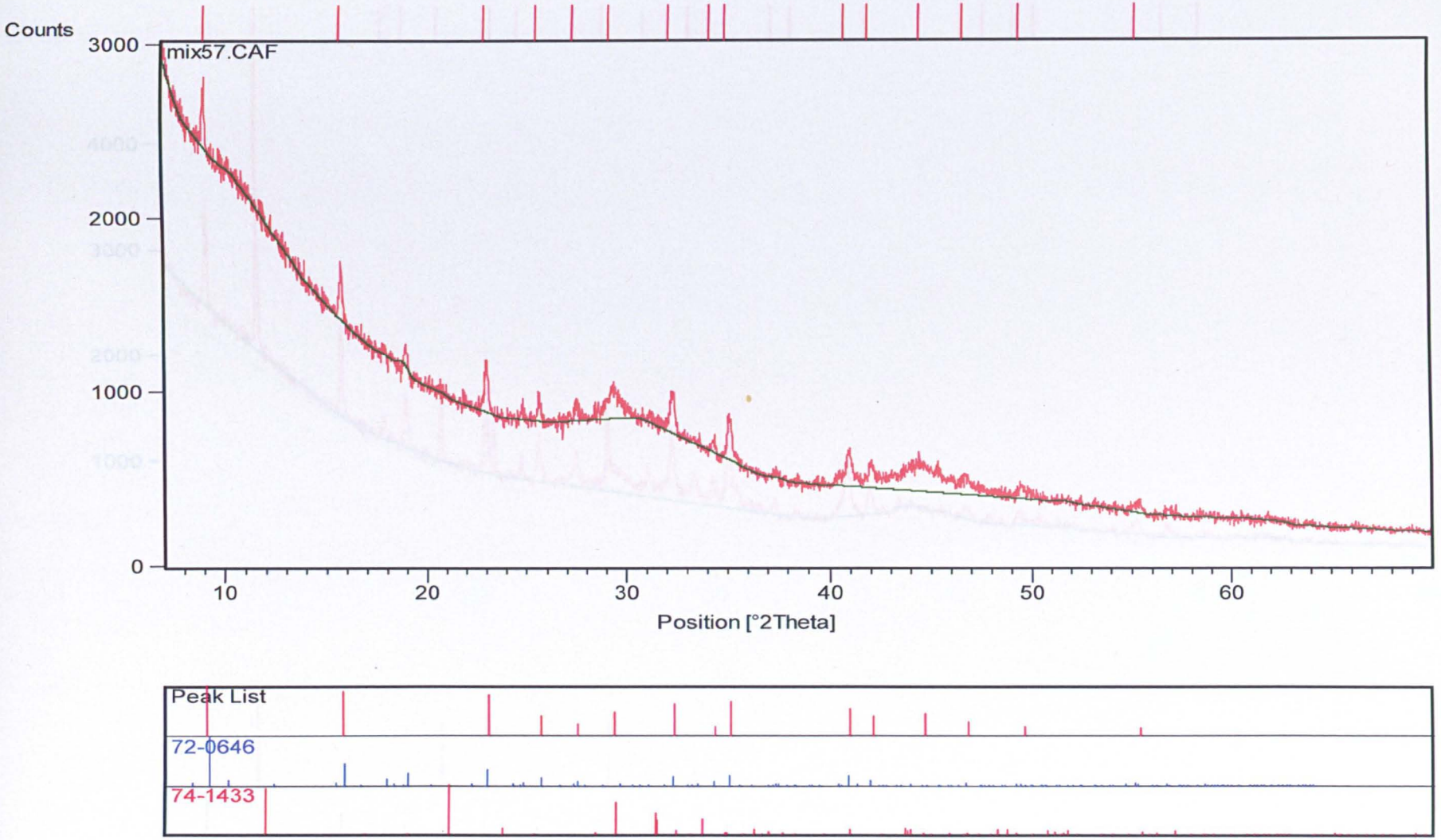
10% RG - 90% GGBS

Date: 15/11/2005 Time: 16:47:22 File: mix66.CAF User: npnh1



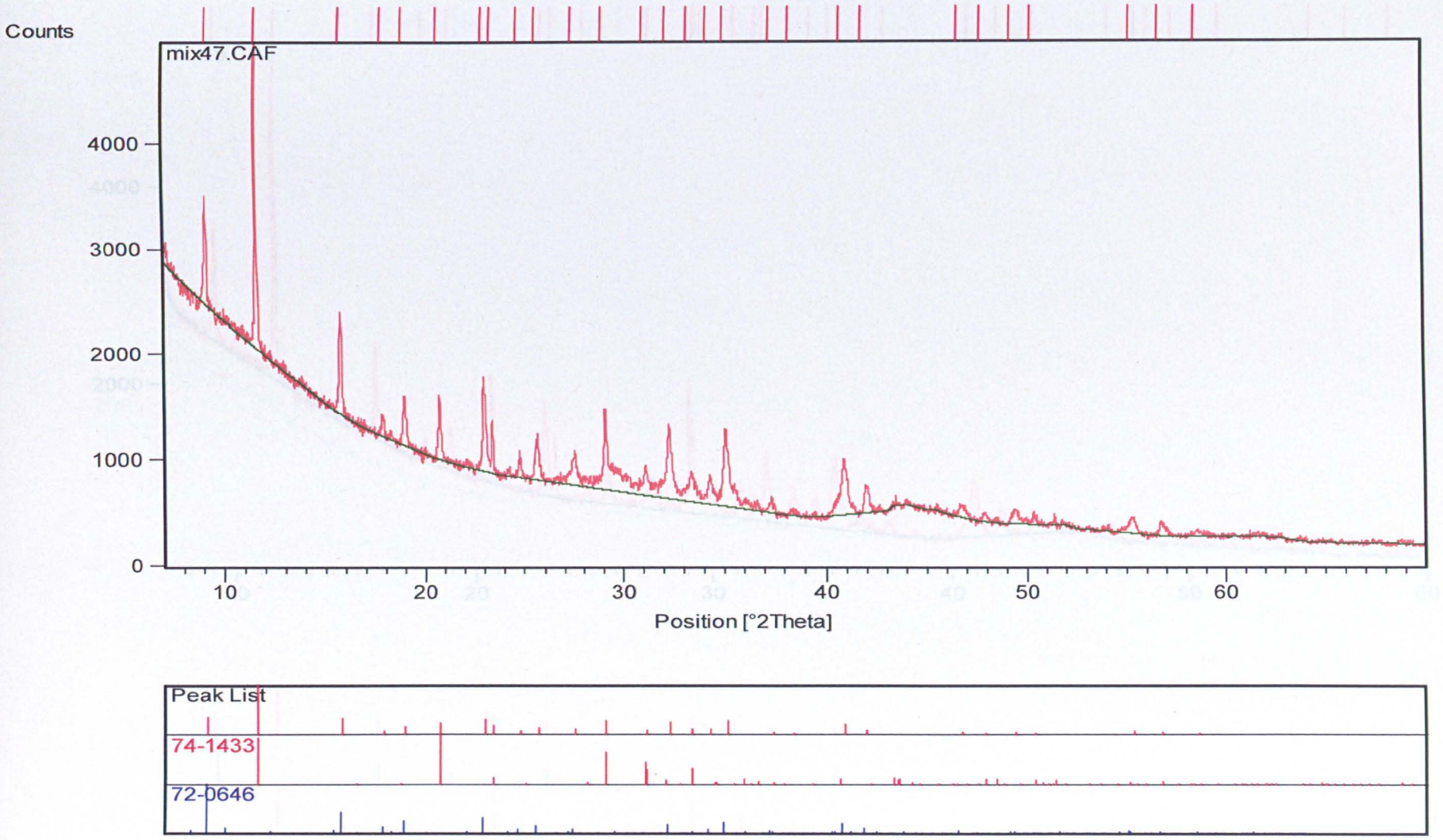
20% RG - 80% GGBS

Date: 15/11/2005 Time: 16:46:50 File: mix57.CAF User: npnh1



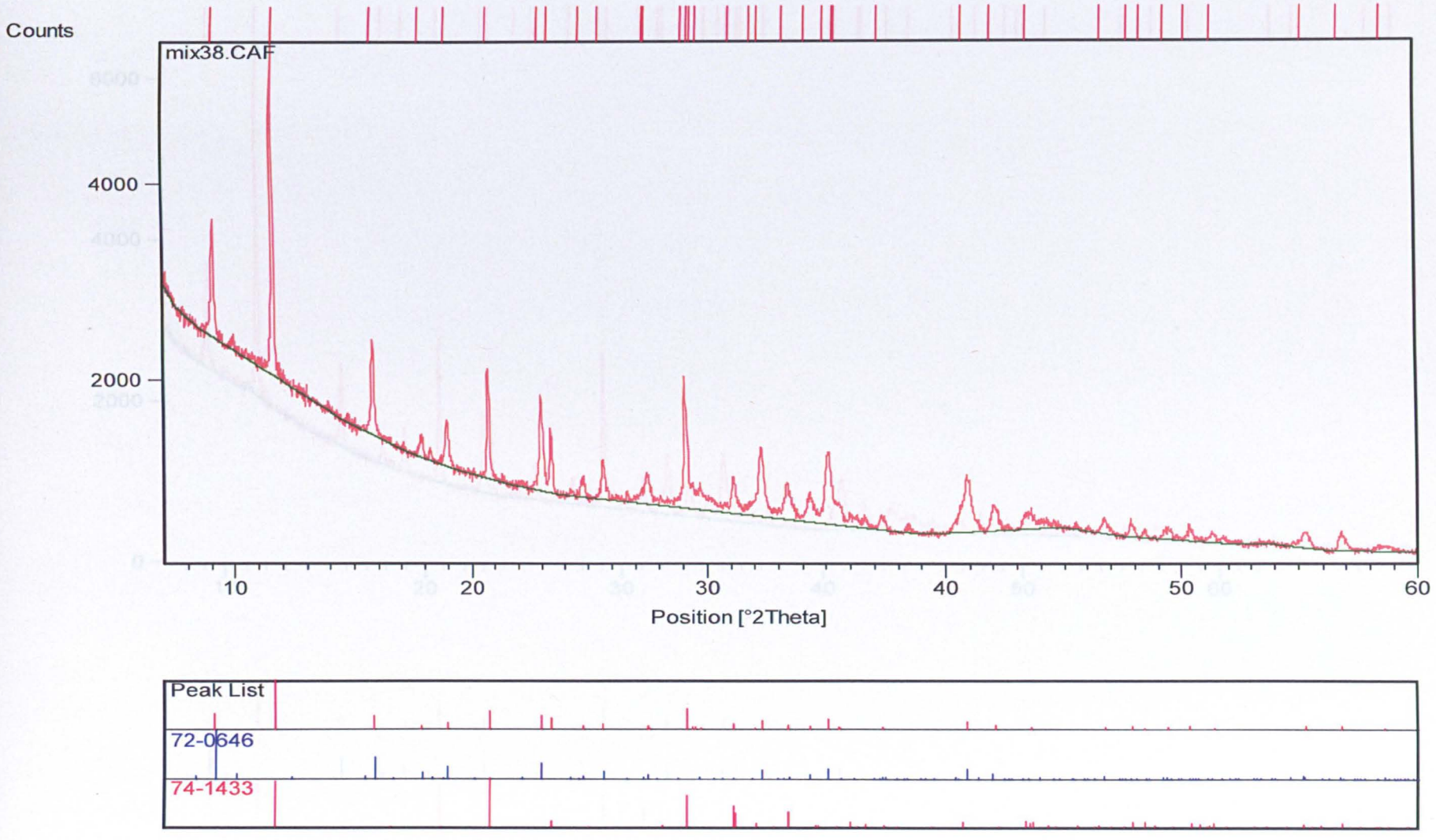
30% RG - 70% GGBS

Date: 15/11/2005 Time: 16:46:18 File: mix47.CAF User: npnh1



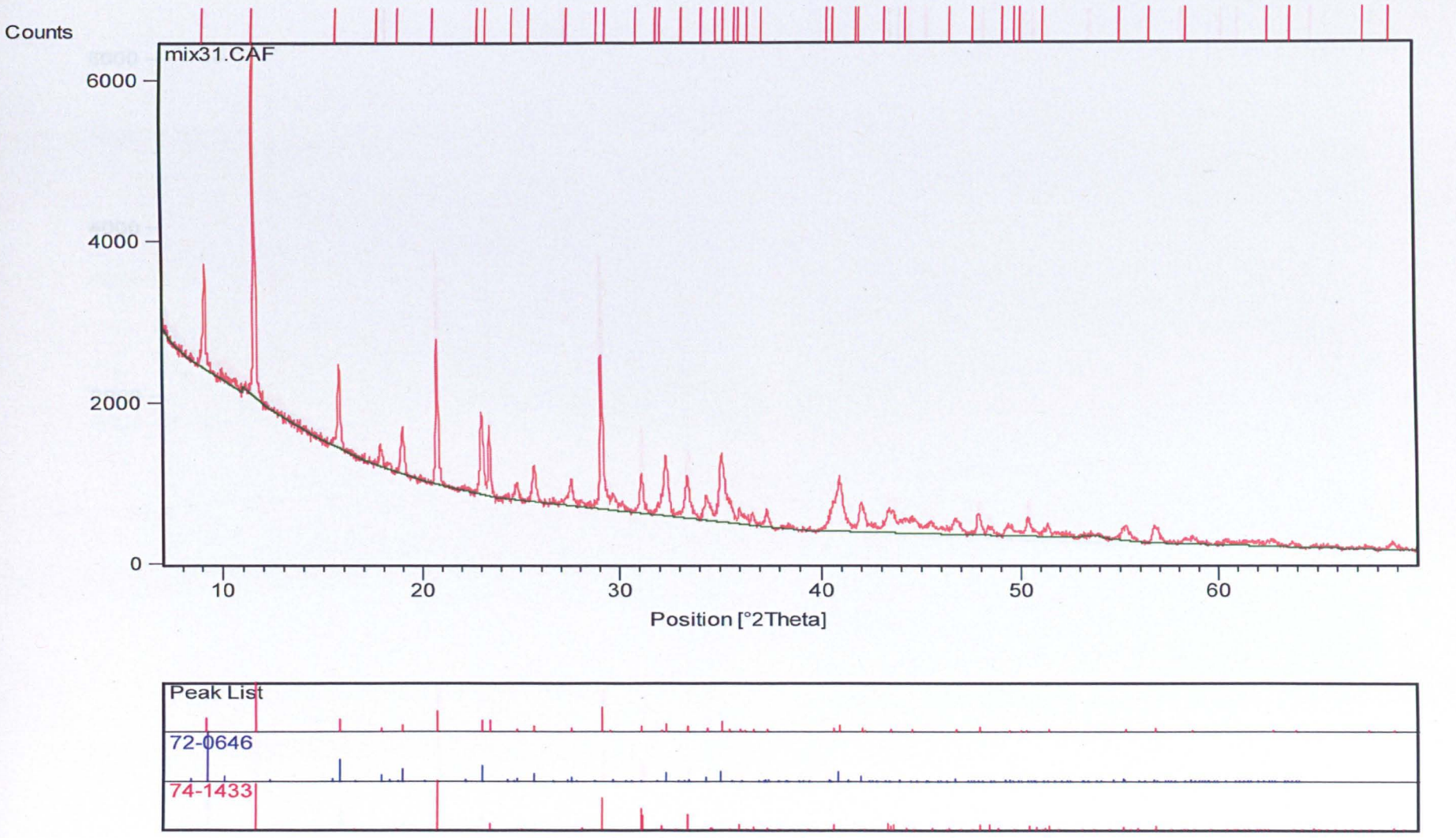
40% RG - 60% GGBS

Date: 15/11/2005 Time: 16:45:48 File: mix38.CAF User: npnh1



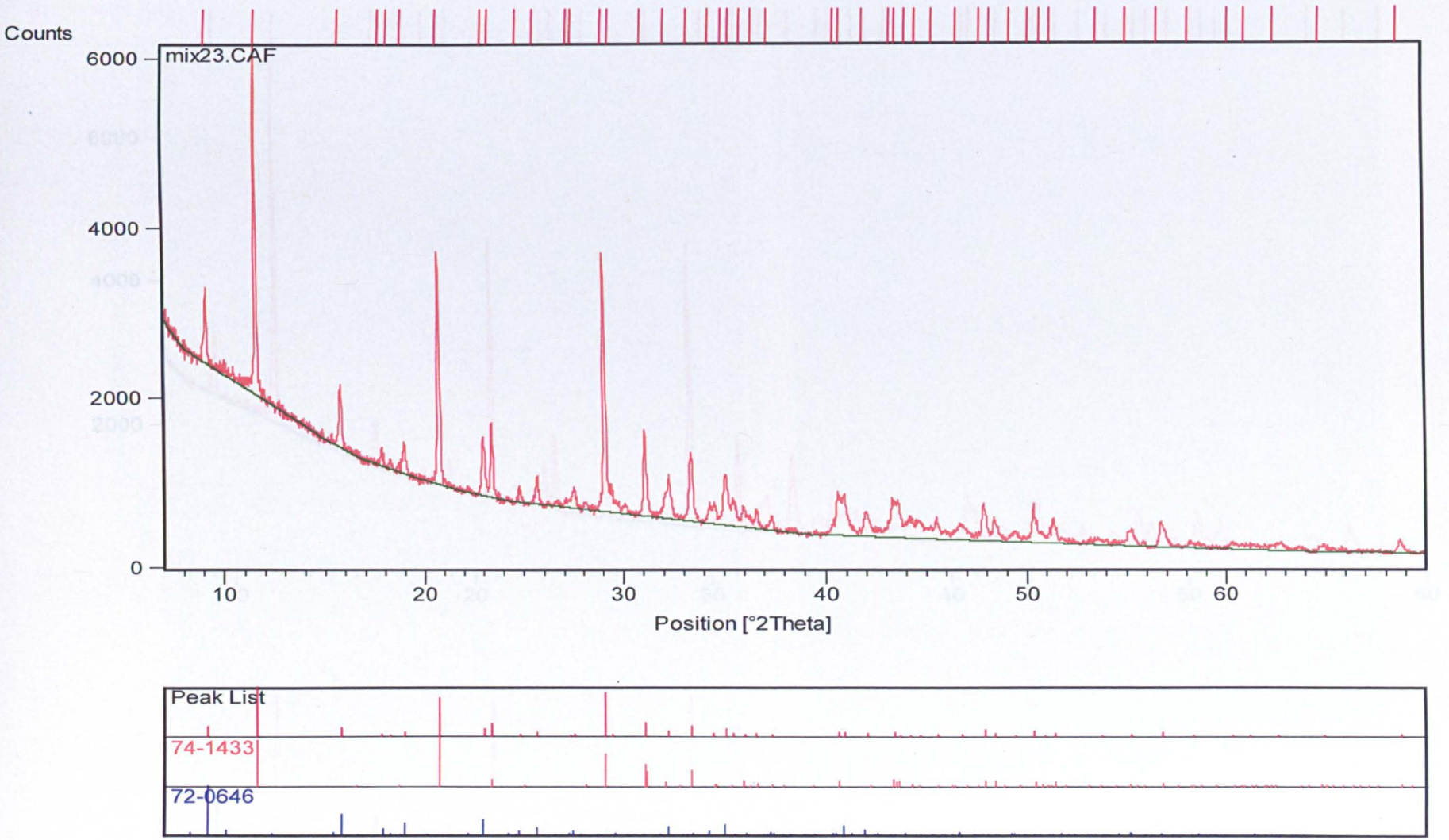
50% RG - 50% GGBS

Date: 15/11/2005 Time: 16:45:14 File: mix31.CAF User: npnh1



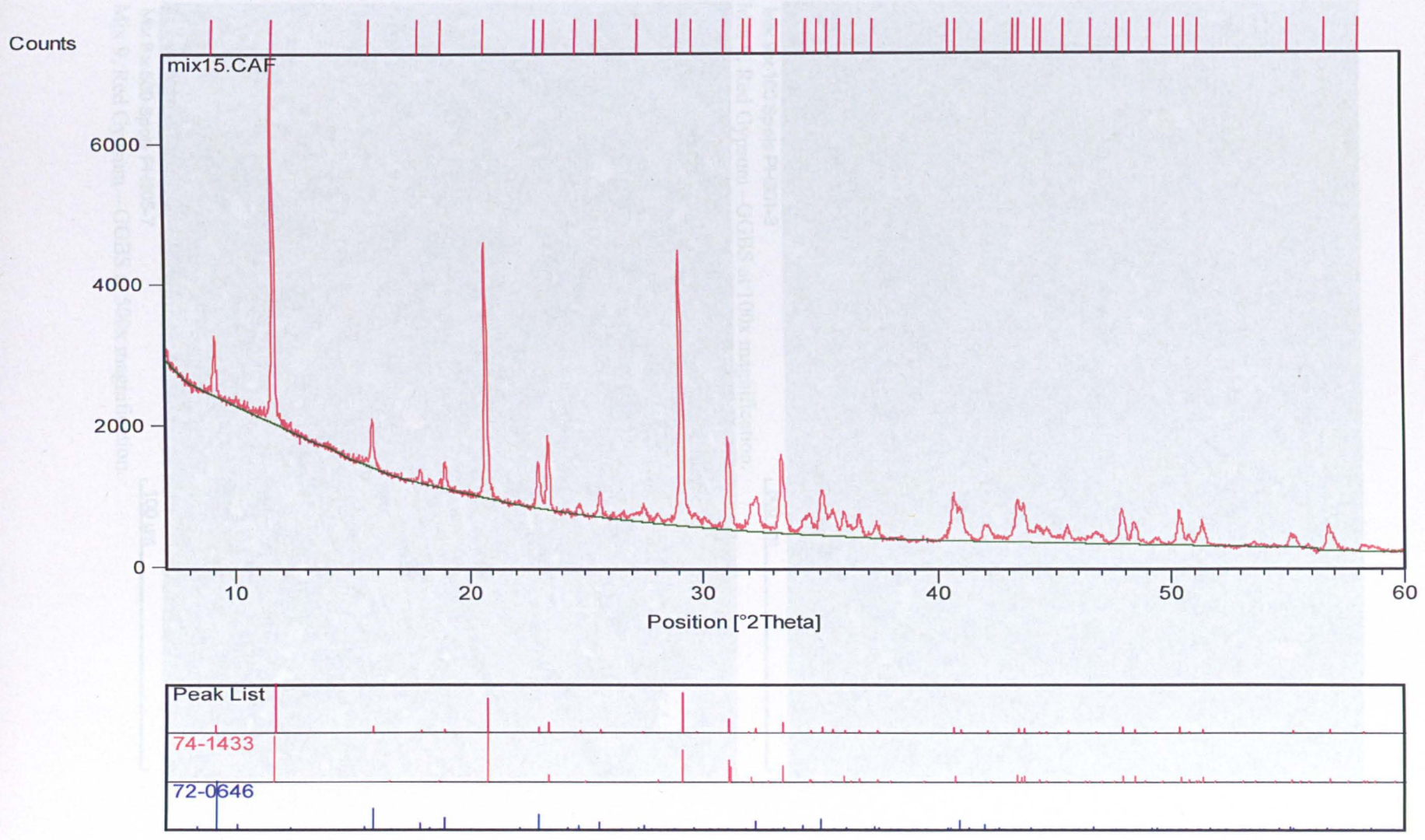
60% RG - 40% GGBS

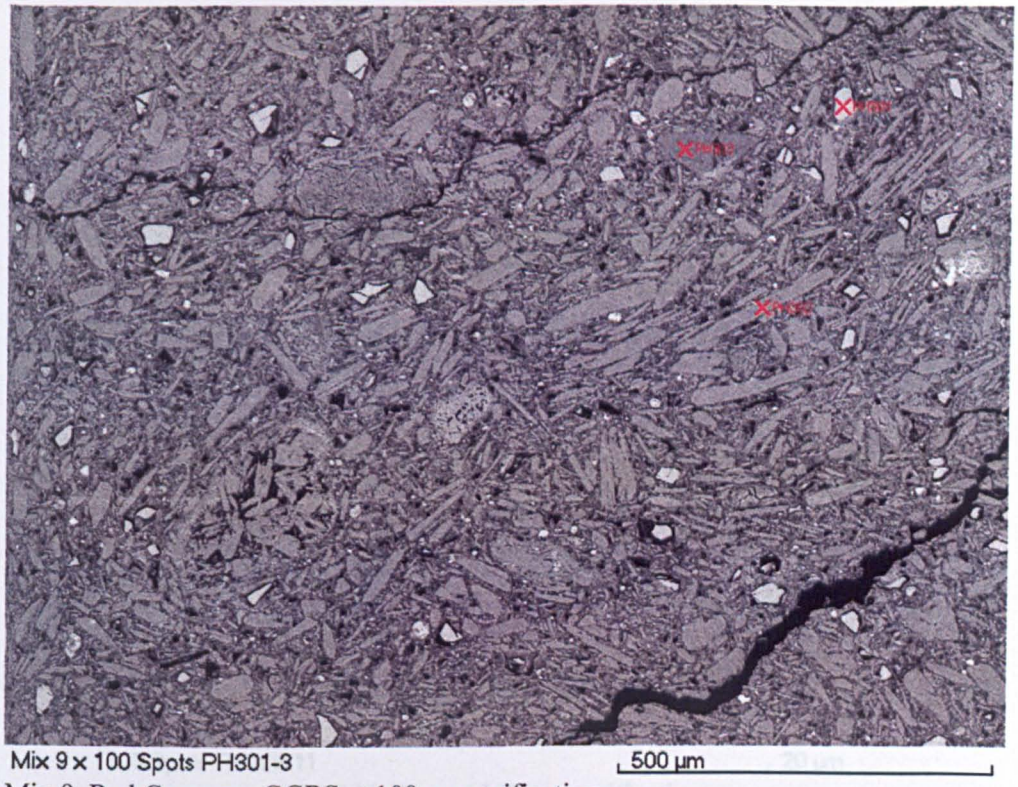
Date: 15/11/2005 Time: 16:44:39 File: mix23.CAF User: npnh1



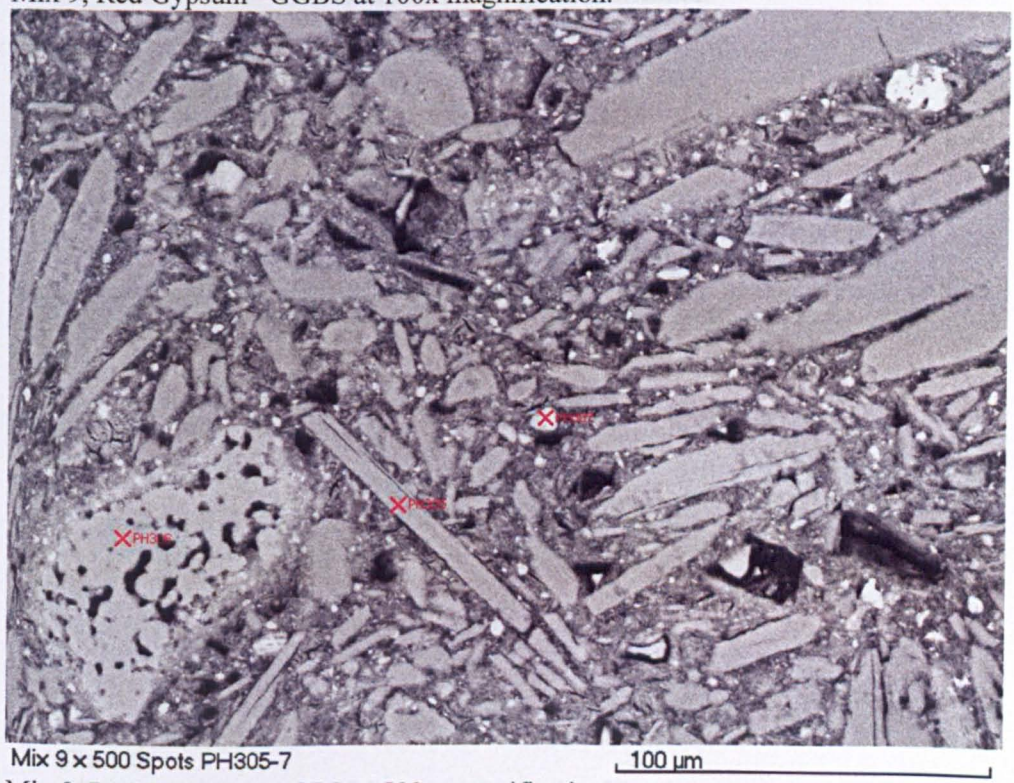
70% RG - 30% GGBS

Date: 15/11/2005 Time: 16:44:07 File: mix15.CAF User: npnh1

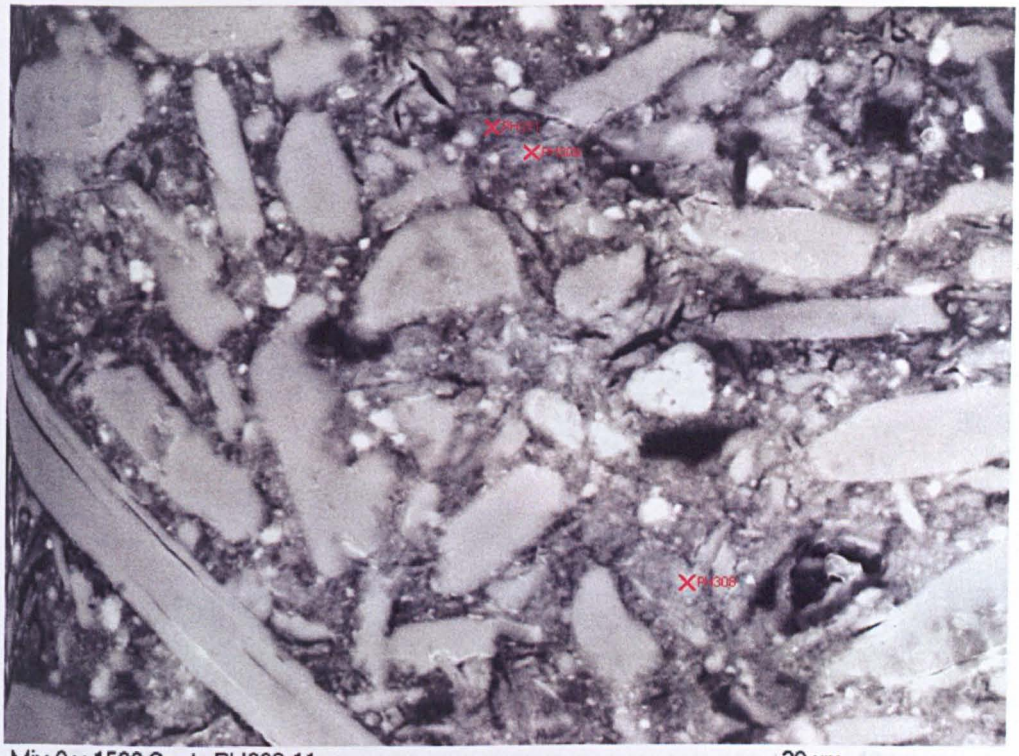




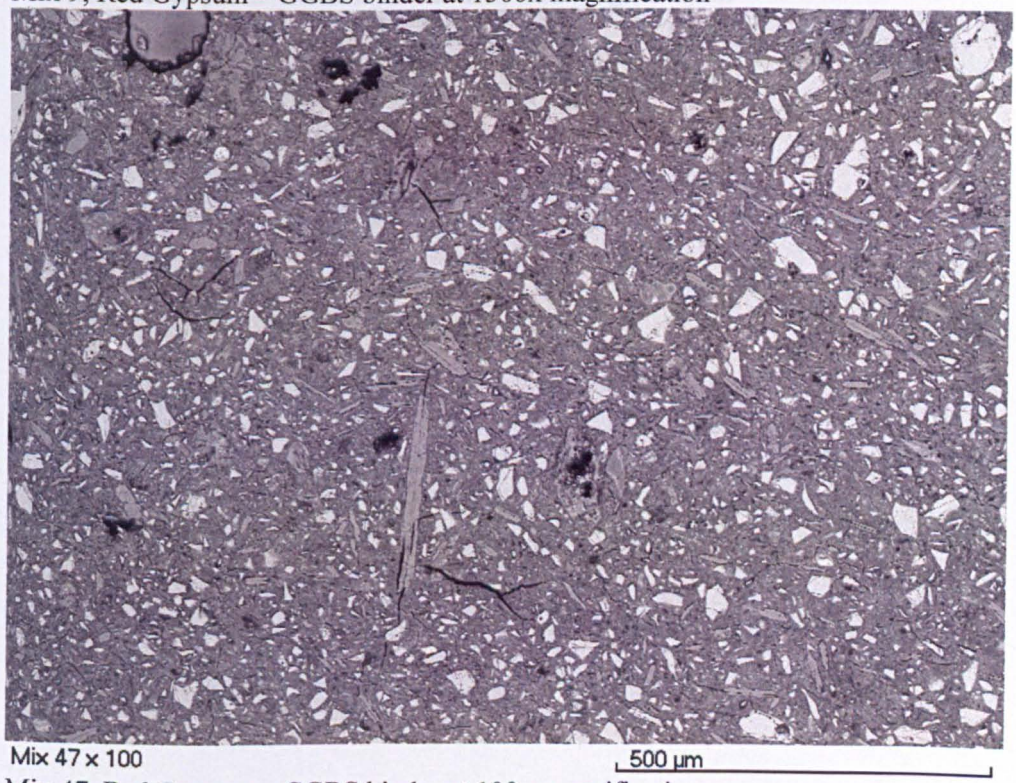

Mix 9, Red Gypsum –GGBS at 100x magnification.



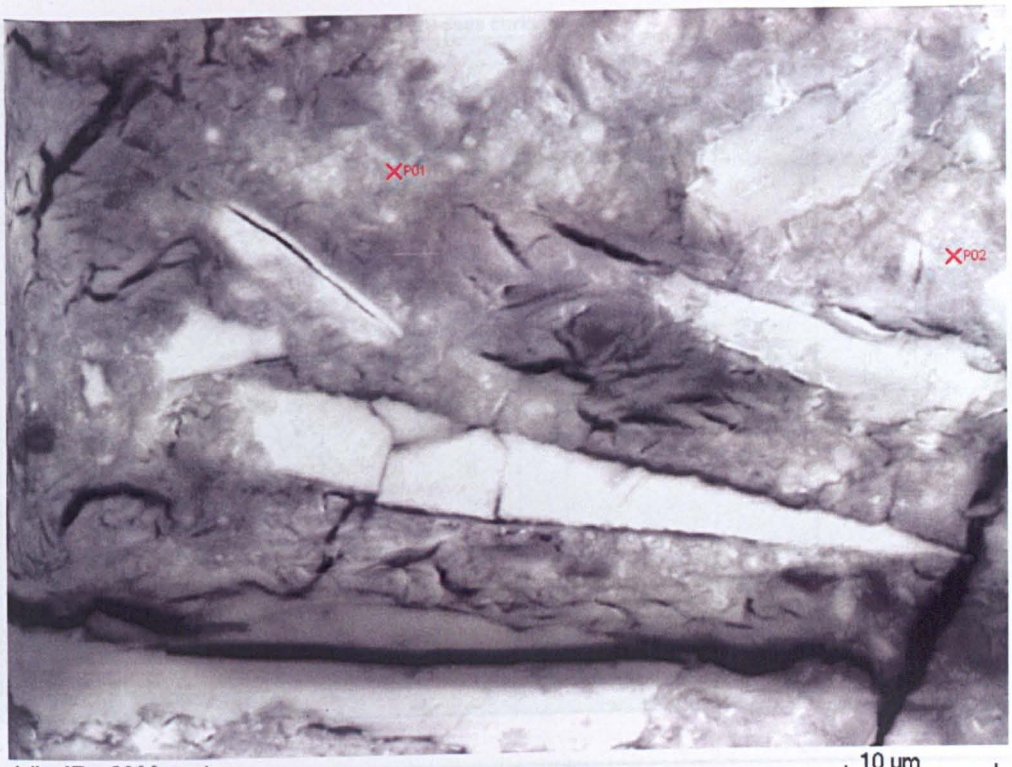

Mix 9, Red Gypsum – GGBS at 500x magnification.




Mix 9, Red Gypsum – GGBS binder at 1500x magnification




Mix 47, Red Gypsum – GGBS binder at 100x magnification.




Mix 47, Red Gypsum – GGBS binder at 2000 x magnification

**Mix 9, 70% Gypsum - 30% GGBS with 2% added lime (28 days curing)**

Template

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| O | 15.999 | N/A | N/A | | N/A | 0 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| Al | 26.982 | Al2O3 | 101.961 | | 0 | 0 | 0 | Ca:Al | 0 | #DIV/0! |
| Si | 28.086 | SiO2 | 60.084 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| P | 30.974 | P4O6 | 219.89 | | 0 | 0 | 0 | Ca:S | 0 | #DIV/0! |
| Cl | 35.453 | ClO2 | 67.451 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| S | 32.065 | SO2 | 64.063 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| K | 39.098 | K2O | 94.195 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Ca | 40.078 | CaO | 56.077 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Ti | 47.867 | TiO2 | 79.865 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Mn | 54.938 | MnO | 70.937 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Fe | 55.845 | Fe2O3 | 159.687 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Ni | 58.6934 | NiO | 74.6924 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| | | | | 0 | 0 | | | | 0 | #DIV/0! |

| Mix 9 x 100 P301 | | | | (a) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 40.94 | N/A | 2.55891 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 4.36 | 7.230012 | 0.179387 | 0.179387 | 1.173132 | 0.179387 | 1.429386 |
| Al | 26.982 | Al2O3 | 101.961 | 6.37 | 12.03565 | 0.236083 | 0.118042 | Ca:Al | 0.70825 | 5.643456 |
| Si | 28.086 | SiO2 | 60.084 | 14.94 | 31.96094 | 0.531938 | 0.531938 | 2.643275 | 1.063875 | 8.477139 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 5.078584 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 3.94 | 7.871767 | 0.122875 | 0.122875 | | 0.245751 | 1.958184 |
| K | 39.098 | K2O | 94.195 | 0.96 | 1.156417 | 0.024554 | 0.012277 | | 0.024554 | 0.195648 |
| Ca | 40.078 | CaO | 56.077 | 25.01 | 34.99391 | 0.624033 | 0.624033 | | 0.624033 | 4.972402 |
| Ti | 47.867 | TiO2 | 79.865 | 0.39 | 0.650706 | 0.008148 | 0.008148 | | 0.016295 | 0.129843 |
| Mn | 54.938 | MnO | 70.937 | 0.47 | 0.606873 | 0.008555 | 0.008555 | | 0.008555 | 0.068168 |
| Fe | 55.845 | Fe2O3 | 159.687 | 2.63 | 3.760201 | 0.047095 | 0.023547 | | 0.141284 | 1.125774 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100.01 | 100.2665 | | | | 3.011984 | 24 |

| Mix 9 x 100 PH302 | | | (b) | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 49.25 | N/A | 3.078317 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 0.42 | 0.696469 | 0.01728 | 0.01728 | 8.954455 | 0.01728 | 0.185569 |
| Al | 26.982 | Al2O3 | 101.961 | 0.62 | 1.171444 | 0.022978 | 0.011489 | Ca:Al | 0.068935 | 0.74027 |
| Si | 28.086 | SiO2 | 60.084 | 1.98 | 4.235787 | 0.070498 | 0.070498 | 27.47242 | 0.140996 | 1.514107 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 1.018191 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 19.88 | 39.71846 | 0.619991 | 0.619991 | | 1.239981 | 13.31577 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | ca;s | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 25.3 | 35.39967 | 0.631269 | 0.631269 | 1.272636 | 0.631269 | 6.779 |
| Ti | 47.867 | TiO2 | 79.865 | 0 | 0 | 0 | 0 | ca;o | 0 | 0 |
| Mn | 54.938 | MnO | 70.937 | 0 | 0 | 0 | 0 | 0.513706 | 0 | 0 |
| Fe | 55.845 | Fe2O3 | 159.687 | 2.54 | 3.631525 | 0.045483 | 0.022742 | | 0.136449 | 1.465284 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 99.99 | 84.85336 | | | | 2.23491 | 24 |

| Mix 9 x 100 PH303 | | | © | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 54.61 | N/A | 3.413338 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 0 | 0 | 0 | 0 | 0.084543 | 0 | 0 |
| Al | 26.982 | Al2O3 | 101.961 | 0 | 0 | 0 | 0 | Ca:Al | 0 | 0 |
| Si | 28.086 | SiO2 | 60.084 | 35.56 | 76.07303 | 1.266111 | 1.266111 | #DIV/0! | 2.532222 | 20.49634 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 1.052846 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 3.26 | 6.513188 | 0.101668 | 0.101668 | | 0.203337 | 1.645852 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 4.29 | 6.002553 | 0.107041 | 0.107041 | | 0.107041 | 0.866414 |
| Ti | 47.867 | TiO2 | 79.865 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Mn | 54.938 | MnO | 70.937 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Fe | 55.845 | Fe2O3 | 159.687 | 2.28 | 3.259794 | 0.040827 | 0.020414 | | 0.122482 | 0.991394 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100 | 91.84856 | | | | 2.965083 | 24 |

| Mix 9 x 500 PH305 | | | (d) | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 49.61 | N/A | 3.100819 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 0.45 | 0.746217 | 0.018515 | 0.018515 | 7.56288 | 0.018515 | 0.197792 |
| Al | 26.982 | Al2O3 | 101.961 | 0.81 | 1.530435 | 0.03002 | 0.01501 | Ca:Al | 0.09006 | 0.96211 |
| Si | 28.086 | SiO2 | 60.084 | 2.26 | 4.834787 | 0.080467 | 0.080467 | 20.27192 | 0.160934 | 1.719258 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 1.049682 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 18.59 | 37.14116 | 0.57976 | 0.57976 | | 1.15952 | 12.38713 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | Ca:s | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 24.39 | 34.1264 | 0.608563 | 0.608563 | 1.311996 | 0.608563 | 6.501272 |
| Ti | 47.867 | TiO2 | 79.865 | 0 | 0 | 0 | 0 | Ca:O | 0 | 0 |
| Mn | 54.938 | MnO | 70.937 | 0 | 0 | 0 | 0 | 0.491635 | 0 | 0 |
| Fe | 55.845 | Fe2O3 | 159.687 | 3.89 | 5.561666 | 0.069657 | 0.034829 | | 0.208971 | 2.232437 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100 | 83.94067 | | | | 2.246563 | 24 |

| Mix 9 x 500 PH306 | | | (e) | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 11.73 | 42.9794 | 0.976605 | 0.976605 | | 1.95321 | 14.47483 |
| O | 15.999 | N/A | N/A | 47.98 | N/A | 2.998937 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 0.46 | 0.762799 | 0.018926 | 0.018926 | 17.2581 | 0.018926 | 0.140258 |
| Al | 26.982 | Al2O3 | 101.961 | 0.54 | 1.02029 | 0.020013 | 0.010007 | Ca:Al | 0.06004 | 0.444944 |
| Si | 28.086 | SiO2 | 60.084 | 1.34 | 2.866644 | 0.047711 | 0.047711 | 41.14227 | 0.095421 | 0.707147 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 10.31334 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 2.56 | 5.114651 | 0.079838 | 0.079838 | | 0.159676 | 1.183323 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 33 | 46.17349 | 0.823394 | 0.823394 | | 0.823394 | 6.102003 |
| Ti | 47.867 | TiO2 | 79.865 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Mn | 54.938 | MnO | 70.937 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Fe | 55.845 | Fe2O3 | 159.687 | 2.38 | 3.402767 | 0.042618 | 0.021309 | | 0.127854 | 0.947498 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 99.99 | 102.32 | | | | 3.238521 | 24 |

| Mix 9 x 500 PH307 | | | | (f) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios (from column (g))** | **(g)xN.o. oxygens** | **normalise (f)*24/Σ(f)** |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 40.27 | N/A | 2.517032 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 1.87 | 3.100945 | 0.076939 | 0.076939 | 1.735743 | 0.076939 | 0.601377 |
| Al | 26.982 | Al2O3 | 101.961 | 2.54 | 4.799143 | 0.094137 | 0.047068 | Ca:Al | 0.28241 | 2.207403 |
| Si | 28.086 | SiO2 | 60.084 | 4.97 | 10.63225 | 0.176956 | 0.176956 | 3.262815 | 0.353913 | 2.766288 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0.93 | 1.769369 | 0.026232 | 0.026232 | 1.712835 | 0.052464 | 0.410073 |
| S | 32.065 | SO2 | 64.063 | 5.75 | 11.48799 | 0.179323 | 0.179323 | | 0.358646 | 2.803287 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 12.31 | 17.22411 | 0.307151 | 0.307151 | | 0.307151 | 2.400783 |
| Ti | 47.867 | TiO2 | 79.865 | 0.32 | 0.533913 | 0.006685 | 0.006685 | | 0.01337 | 0.104507 |
| Mn | 54.938 | MnO | 70.937 | 1.36 | 1.756058 | 0.024755 | 0.024755 | | 0.024755 | 0.193494 |
| Fe | 55.845 | Fe2O3 | 159.687 | 29.8 | 42.60608 | 0.53362 | 0.26681 | | 1.60086 | 12.51279 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100.12 | 93.90985 | | | | 3.070509 | 24 |

| Mix 9 x 1500 PH308 | | | | (g) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios (from column (g))** | **(g)xN.o. oxygens** | **normalise (f)*24/Σ(f)** |
| C | 12.011 | CO2 | 44.009 | 13.34 | 48.87853 | 1.110649 | 1.110649 | | 2.221297 | 11.56845 |
| O | 15.999 | N/A | N/A | 44.3 | N/A | 2.768923 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 1.35 | 2.23865 | 0.055544 | 0.055544 | 0.403651 | 0.055544 | 0.289272 |
| Al | 26.982 | Al2O3 | 101.961 | 1.98 | 3.741064 | 0.073382 | 0.036691 | Ca:Al | 0.220147 | 1.146518 |
| Si | 28.086 | SiO2 | 60.084 | 15 | 32.0893 | 0.534074 | 0.534074 | 2.937762 | 1.068148 | 5.562883 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0.6 | 1.141528 | 0.016924 | 0.016924 | 1.681888 | 0.033848 | 0.176277 |
| S | 32.065 | SO2 | 64.063 | 4.11 | 8.211412 | 0.128177 | 0.128177 | | 0.256354 | 1.335086 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | ca:o | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 8.64 | 12.08906 | 0.21558 | 0.21558 | 0.195034 | 0.21558 | 1.122732 |
| Ti | 47.867 | TiO2 | 79.865 | 1.48 | 2.469346 | 0.030919 | 0.030919 | ca:si | 0.061838 | 0.322051 |
| Mn | 54.938 | MnO | 70.937 | 0.48 | 0.619785 | 0.008737 | 0.008737 | 0.93331 | 0.008737 | 0.045503 |
| Fe | 55.845 | Fe2O3 | 159.687 | 8.69 | 12.42439 | 0.155609 | 0.077805 | | 0.466828 | 2.431226 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 99.97 | 123.9031 | | | | 4.60832 | 24 |

| Mix 9 x 1500 PH309 | | | | (h) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios (from column (g))** | **(g)xN.o. oxygens** | **normalise (f)*24/Σ(f)** |
| C | 12.011 | CO2 | 44.009 | 10.19 | 37.33675 | 0.848389 | 0.848389 | | 1.696778 | 10.68551 |
| O | 15.999 | N/A | N/A | 45.87 | N/A | 2.867054 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 1.35 | 2.23865 | 0.055544 | 0.055544 | 2.849521 | 0.055544 | 0.349791 |
| Al | 26.982 | Al2O3 | 101.961 | 2.16 | 4.081161 | 0.080053 | 0.040027 | Ca:Al | 0.24016 | 1.512416 |
| Si | 28.086 | SiO2 | 60.084 | 4.23 | 9.049182 | 0.150609 | 0.150609 | 5.360963 | 0.301218 | 1.896928 |
| P | 30.974 | P4O6 | 219.89 | | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 1.328293 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 10.36 | 20.69835 | 0.323094 | 0.323094 | ca:si | 0.646187 | 4.069386 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | 4.066194 | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 17.2 | 24.06618 | 0.429163 | 0.429163 | ca;o | 0.429163 | 2.702669 |
| Ti | 47.867 | TiO2 | 79.865 | 0.37 | 0.617337 | 0.00773 | 0.00773 | 0.374973 | 0.01546 | 0.097357 |
| Mn | 54.938 | MnO | 70.937 | 0.5 | 0.64561 | 0.009101 | 0.009101 | ca:s | 0.009101 | 0.057315 |
| Fe | 55.845 | Fe2O3 | 159.687 | 7.77 | 11.10903 | 0.139135 | 0.069568 | 0.875341 | 0.417405 | 2.628624 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100 | 109.8423 | | | | 3.811016 | 24 |

| Mix 9 x 1500 PH311 | | | | (i) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios (from column (g))** | **(g)xN.o. oxygens** | **normalise (f)*24/Σ(f)** |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 46.95 | N/A | 2.934558 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 1.63 | 2.702963 | 0.067064 | 0.067064 | 2.789579 | 0.067064 | 0.621151 |
| Al | 26.982 | Al2O3 | 101.961 | 3.35 | 6.329578 | 0.124157 | 0.062078 | Ca:Al | 0.372471 | 3.449827 |
| Si | 28.086 | SiO2 | 60.084 | 5.17 | 11.06011 | 0.184077 | 0.184077 | 4.135887 | 0.368155 | 3.409856 |
| P | 30.974 | P4O6 | 219.89 | 0 | 0 | 0 | 0 | Ca:S | 0 | 0 |
| Cl | 35.453 | ClO2 | 67.451 | 0 | 0 | 0 | 0 | 1.404892 | 0 | 0 |
| S | 32.065 | SO2 | 64.063 | 11.72 | 23.41551 | 0.365508 | 0.365508 | ca:o | 0.731015 | 6.770672 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | 0.438339 | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 20.58 | 28.79547 | 0.513499 | 0.513499 | Ca;s | 0.513499 | 4.756031 |
| Ti | 47.867 | TiO2 | 79.865 | 0.45 | 0.750815 | 0.009401 | 0.009401 | 1.755973 | 0.018802 | 0.174145 |
| Mn | 54.938 | MnO | 70.937 | 0.72 | 0.929678 | 0.013106 | 0.013106 | ca:si | 0.013106 | 0.121385 |
| Fe | 55.845 | Fe2O3 | 159.687 | 9.44 | 13.49669 | 0.169039 | 0.08452 | 3.980658 | 0.507118 | 4.696933 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100.01 | 87.48081 | | | | 2.591229 | 24 |

**Mix 47, 30% red gypsum, 70% GGBS + 2% Lime 28 days curing**

**Template**

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios (from column (g)) | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| O | 15.999 | N/A | N/A | | N/A | 0 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| Al | 26.982 | Al2O3 | 101.961 | | 0 | 0 | 0 | Ca:Al | 0 | #DIV/0! |
| Si | 28.086 | SiO2 | 60.084 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| S | 32.065 | SO2 | 64.063 | | 0 | 0 | 0 | Ca:S | 0 | #DIV/0! |
| K | 39.098 | K2O | 94.195 | | 0 | 0 | 0 | #DIV/0! | 0 | #DIV/0! |
| Ca | 40.078 | CaO | 56.077 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Ti | 47.867 | TiO2 | 79.865 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Mn | 54.938 | MnO | 70.937 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Fe | 55.845 | Fe2O3 | 159.687 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| Ni | 58.6934 | NiO | 74.6924 | | 0 | 0 | 0 | | 0 | #DIV/0! |
| | | | | 0 | 0 | | | | 0 | #DIV/0! |

**Mix 47 x 500 P01**

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 40.44 | N/A | 2.527658 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 4.53 | 7.5119161 | 0.1863814 | 0.1863814 | 1.1529442 | 0.1863814 | 1.4700034 |
| Al | 26.982 | Al2O3 | 101.961 | 7.31 | 13.811706 | 0.2709214 | 0.1354607 | Ca:Al | 0.8127641 | 6.4103281 |
| Si | 28.086 | SiO2 | 60.084 | 16.01 | 34.249977 | 0.5700349 | 0.5700349 | 2.4258642 | 1.1400698 | 8.9918116 |
| S | 32.065 | SO2 | 64.063 | 2.25 | 4.4952986 | 0.07017 | 0.07017 | Ca:S | 0.1403399 | 1.1068711 |
| K | 39.098 | K2O | 94.195 | 0.88 | 1.0600491 | 0.0225075 | 0.0112538 | 9.3660928 | 0.0225075 | 0.1775186 |
| Ca | 40.078 | CaO | 56.077 | 26.34 | 36.854838 | 0.6572184 | 0.6572184 | | 0.6572184 | 5.1835285 |
| Ti | 47.867 | TiO2 | 79.865 | 0.34 | 0.5672823 | 0.007103 | 0.007103 | | 0.014206 | 0.112044 |
| Mn | 54.938 | MnO | 70.937 | 0.57 | 0.7359949 | 0.0103753 | 0.0103753 | | 0.0103753 | 0.081831 |
| Fe | 55.845 | Fe2O3 | 159.687 | 1.1 | 1.5727075 | 0.0196974 | 0.0098487 | | 0.0590921 | 0.4660638 |
| Ni | 58.6934 | NiO | 74.6924 | 0.23 | 0.2926948 | 0.0039187 | 0.0039187 | | 0.0039187 | 0.0309068 |
| | | | | 100 | 100.85977 | | | | 3.0429546 | 24 |

**Mix 47 x 500 P02**

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios** | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 48.25 | N/A | 3.0158135 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 0.7 | 1.1607817 | 0.0288007 | 0.0288007 | 5.2063642 | 0.0288007 | 0.3039112 |
| Al | 26.982 | Al2O3 | 101.961 | 1.22 | 2.3051001 | 0.0452153 | 0.0226077 | Ca:Al | 0.135646 | 1.431368 |
| Si | 28.086 | SiO2 | 60.084 | 3.68 | 7.8725742 | 0.1310261 | 0.1310261 | 15.087135 | 0.2620523 | 2.7652364 |
| S | 32.065 | SO2 | 64.063 | 17.88 | 35.72264 | 0.5576173 | 0.5576173 | Ca:S | 1.1152347 | 11.768215 |
| K | 39.098 | K2O | 94.195 | 0 | 0 | 0 | 0 | 1.2233654 | 0 | 0 |
| Ca | 40.078 | CaO | 56.077 | 27.34 | 38.254034 | 0.6821698 | 0.6821698 | | 0.6821698 | 7.1984139 |
| Ti | 47.867 | TiO2 | 79.865 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Mn | 54.938 | MnO | 70.937 | 0 | 0 | 0 | 0 | | 0 | 0 |
| Fe | 55.845 | Fe2O3 | 159.687 | 0.94 | 1.34395 | 0.0168323 | 0.0084162 | | 0.0504969 | 0.5328551 |
| Ni | 58.6934 | NiO | 74.6924 | 0 | 0 | 0 | 0 | | 0 | 0 |
| | | | | 100.01 | 86.65908 | | | | 2.2744003 | 24 |

**Mix 47 x 500 P03**

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| **Element** | **atomic weight** | **oxide** | **atomic weight of oxide** | **weight % of element** | **weight % of oxide** | **atomic proportions** | **concrete** | **Ratios** | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 39.49 | N/A | 2.4682793 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 4.59 | 7.6114116 | 0.18885 | 0.18885 | 1.1431725 | 0.18885 | 1.4775647 |
| Al | 26.982 | Al2O3 | 101.961 | 6.8 | 12.848099 | 0.2520199 | 0.1260099 | Ca:Al | 0.7560596 | 5.9154185 |
| Si | 28.086 | SiO2 | 60.084 | 15.73 | 33.650976 | 0.5600655 | 0.5600655 | 2.5404803 | 1.120131 | 8.7639173 |
| S | 32.065 | SO2 | 64.063 | 3.43 | 6.852833 | 0.1069702 | 0.1069702 | Ca:S | 0.2139404 | 1.6738723 |
| K | 39.098 | K2O | 94.195 | 1.1 | 1.3250614 | 0.0281344 | 0.0140672 | 5.985325 | 0.0281344 | 0.2201241 |
| Ca | 40.078 | CaO | 56.077 | 25.66 | 35.903384 | 0.6402515 | 0.6402515 | | 0.6402515 | 5.0093348 |
| Ti | 47.867 | TiO2 | 79.865 | 0.5 | 0.8342386 | 0.0104456 | 0.0104456 | | 0.0208912 | 0.1634531 |
| Mn | 54.938 | MnO | 70.937 | 0.67 | 0.8651169 | 0.0121956 | 0.0121956 | | 0.0121956 | 0.0954182 |
| Fe | 55.845 | Fe2O3 | 159.687 | 1.62 | 2.3161692 | 0.0290089 | 0.0145044 | | 0.0870266 | 0.680897 |
| Ni | 58.6934 | NiO | 74.6924 | 0.42 | 0.5344861 | 0.0071558 | 0.0071558 | | 0.0071558 | 0.0559873 |
| | | | | 100.01 | 102.20729 | | | | 3.0674804 | 24 |

Mix 47 x 500 P01

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 45.24 | N/A | 2.8276767 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 3.46 | 5.7375783 | 0.1423575 | 0.1423575 | 1.3362766 | 0.1423575 | 1.2571117 |
| Al | 26.982 | Al2O3 | 101.961 | 4.85 | 9.1637175 | 0.1797495 | 0.0898747 | Ca:Al | 0.5392484 | 4.7619217 |
| Si | 28.086 | SiO2 | 60.084 | 11.27 | 24.109759 | 0.4012675 | 0.4012675 | 2.9830654 | 0.8025351 | 7.0869181 |
| S | 32.065 | SO2 | 64.063 | 6.23 | 12.446982 | 0.1942928 | 0.1942928 | Ca:S | 0.3885857 | 3.4314699 |
| K | 39.098 | K2O | 94.195 | 0.9 | 1.0841411 | 0.0230191 | 0.0115095 | 2.7597743 | 0.0230191 | 0.2032738 |
| Ca | 40.078 | CaO | 56.077 | 21.49 | 30.068734 | 0.5362044 | 0.5362044 | | 0.5362044 | 4.7350413 |
| Ti | 47.867 | TiO2 | 79.865 | 0.52 | 0.8676082 | 0.0108634 | 0.0108634 | | 0.0217269 | 0.1918627 |
| Mn | 54.938 | MnO | 70.937 | 0.61 | 0.7876437 | 0.0111034 | 0.0111034 | | 0.0111034 | 0.0980506 |
| Fe | 55.845 | Fe2O3 | 159.687 | 4.71 | 6.7340475 | 0.0843406 | 0.0421703 | | 0.2530218 | 2.2343503 |
| Ni | 58.6934 | NiO | 74.6924 | 0.73 | 0.9289878 | 0.0124375 | 0.0124375 | | 0.0124375 | 0.1098315 |
| | | | | 100.01 | 91.000211 | | | | 2.7178022 | 24 |

Mix 47 x 500 P02

| (a) | (b) | © | (d) | (e) | (f) | (g) | (h) | (i) | (f) | (g) |
|---|---|---|---|---|---|---|---|---|---|---|
| Element | atomic weight | oxide | atomic weight of oxide | weight % of element | weight % of oxide | atomic proportions | concrete | Ratios | (g)xN.o. oxygens | normalise (f)*24/Σ(f) |
| C | 12.011 | CO2 | 44.009 | 0 | 0 | 0 | 0 | | 0 | 0 |
| O | 15.999 | N/A | N/A | 44.77 | N/A | 2.7982999 | N/A | Ca:Si | N/A | N/A |
| Mg | 24.305 | MgO | 40.304 | 3.2 | 5.3064308 | 0.1316602 | 0.1316602 | 1.4377487 | 0.1316602 | 1.178766 |
| Al | 26.982 | Al2O3 | 101.961 | 4.2 | 7.9355904 | 0.1556593 | 0.0778297 | Ca:Al | 0.466978 | 4.180899 |
| Si | 28.086 | SiO2 | 60.084 | 11.04 | 23.617723 | 0.3930784 | 0.3930784 | 3.630672 | 0.7861568 | 7.0385377 |
| S | 32.065 | SO2 | 64.063 | 6.69 | 13.366021 | 0.2086387 | 0.2086387 | Ca:S | 0.4172774 | 3.7359249 |
| K | 39.098 | K2O | 94.195 | 1.03 | 1.2407393 | 0.0263441 | 0.013172 | 2.7087398 | 0.0263441 | 0.2358609 |
| Ca | 40.078 | CaO | 56.077 | 22.65 | 31.691802 | 0.565148 | 0.565148 | | 0.565148 | 5.0598242 |
| Ti | 47.867 | TiO2 | 79.865 | 0.51 | 0.8509234 | 0.0106545 | 0.0106545 | | 0.021309 | 0.1907819 |
| Mn | 54.938 | MnO | 70.937 | 0.7 | 0.9038534 | 0.0127416 | 0.0127416 | | 0.0127416 | 0.1140771 |
| Fe | 55.845 | Fe2O3 | 159.687 | 4.71 | 6.7340475 | 0.0843406 | 0.0421703 | | 0.2530218 | 2.2653282 |
| Ni | 58.6934 | NiO | 74.6924 | 0.51 | 0.6490189 | 0.0086892 | 0.0086892 | | 0.0086892 | 0.0777954 |
| | | | | 100.01 | 91.647131 | | | | 2.6806368 | 24 |

# Appendix C

# Soil mixing testing data

# Appendix C Contents



## Sample Notation for XRD plots

PH SS G D = silty sand, red gypsum – GGBS binder, dry cured
PH SS G W = silty sand, red gypsum – GGBS binder, soaked curing
PH SS O D = silty sand, Portland cement binder, dry cured
PH SS O W = silty sand, Portland cement binder, soaked curing
PH GT G D = Glacial Till, red gypsum – GGBS binder, dry cured
PH GT G D = Glacial Till, red gypsum – GGBS binder, soaked curing
PH GT G D = Glacial Till, Portland cement binder, dry cured
PH GT G D = Glacial Till, Portland cement binder, soaked curing
PH LC G D = London Clay, red gypsum – GGBS binder, soaked curing
PH LC O D = London Clay, Portland cement binder, dry cured
PH LC O W = London Clay, Portland cement binder, soaked curing

## Sample Notation for SEM images and point analysis data

A1 = silty sand, Portland cement binder, dry cured
A2 = silty sand, Portland cement binder, soaked curing
A3 = silty sand, red gypsum – GGBS binder, dry cured
A4 = silty sand, red gypsum – GGBS binder, soaked curing
A5 = Glacial Till, Portland cement binder, dry cured
A6 = Glacial Till, Portland cement binder, soaked curing
A7 = Glacial Till, red gypsum – GGBS binder, dry cured
A8 = Glacial Till, red gypsum – GGBS binder, soaked curing
A9 = London Clay, Portland cement binder, dry cured
A10 = London Clay, Portland cement binder, soaked
A11 = London Clay, red gypsum – GGBS binder, soaked curing

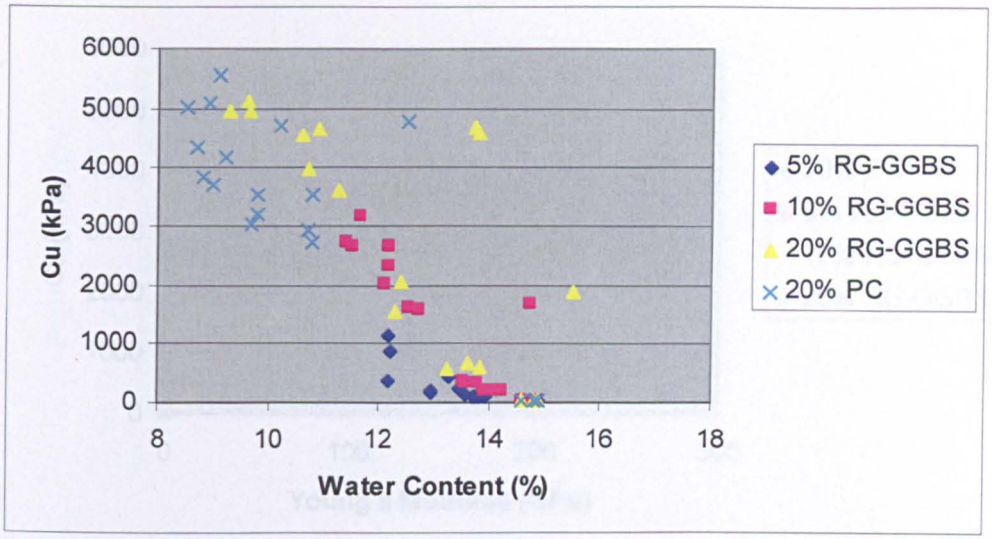



Figure C1 Un-soaked Silty sand, Shear strength against water content.

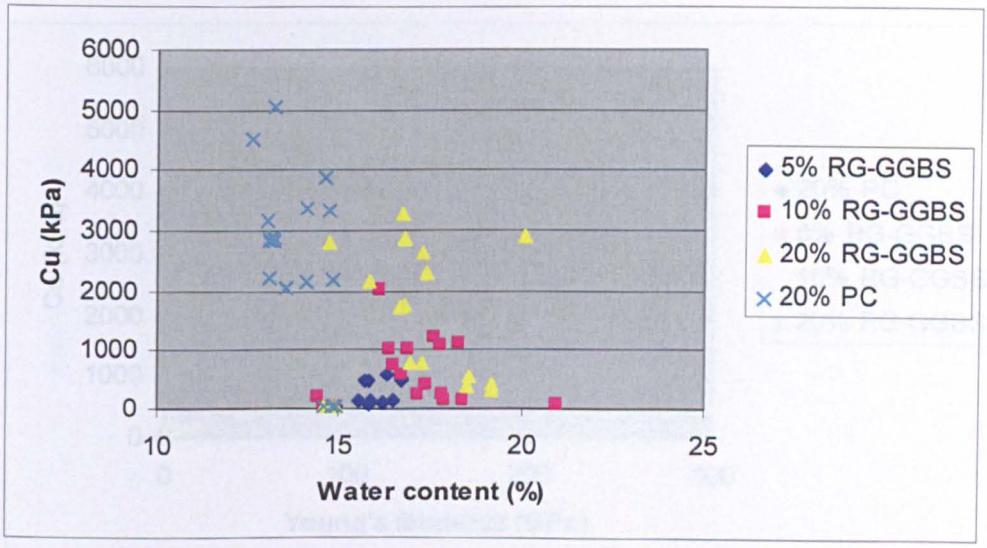



Figure C2 Soaked Silty sand, Shear strength against water content.

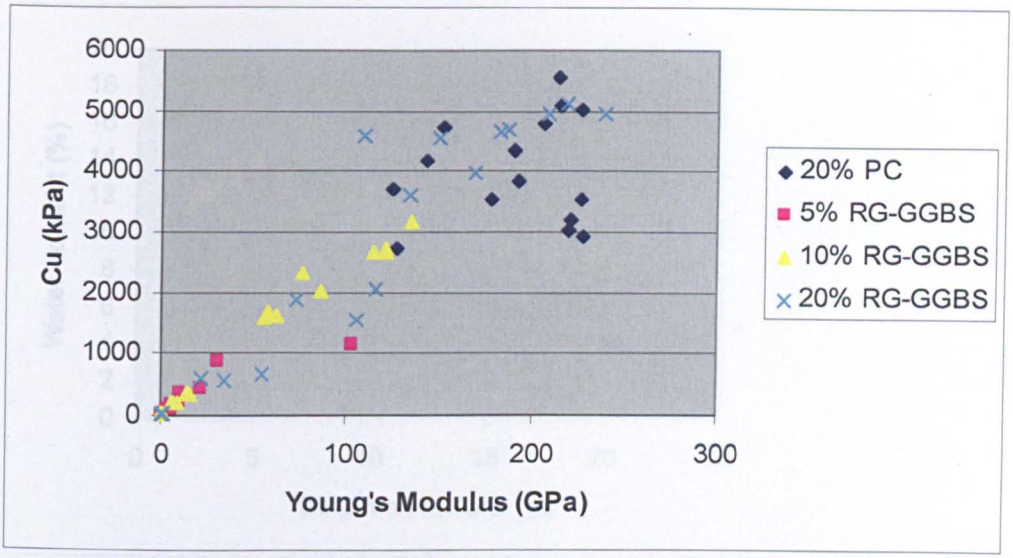



Figure C3 Un-soaked Silty sand, Shear strength against stiffness.

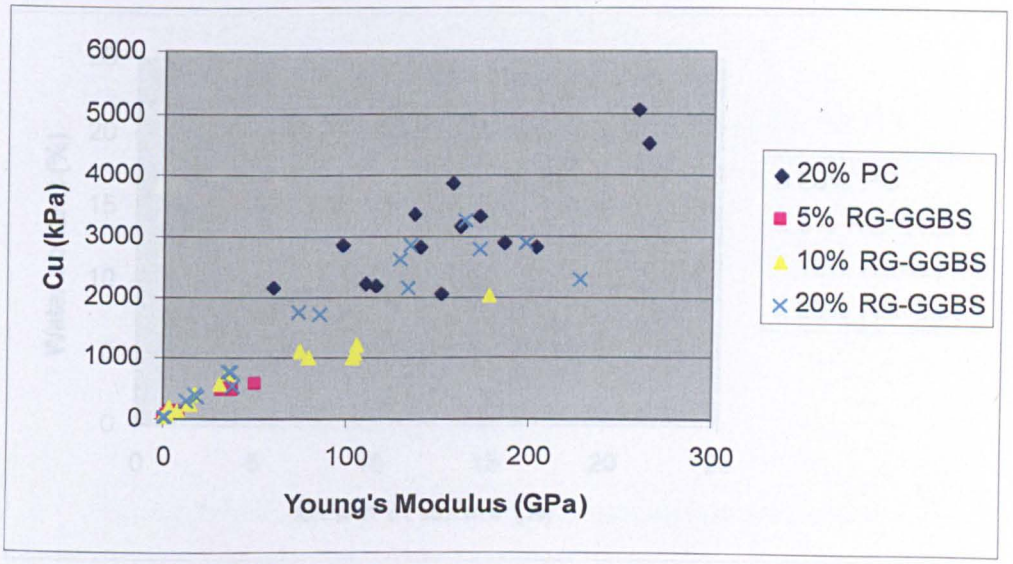



Figure C4 Soaked silty sand, shear strength against stiffness.

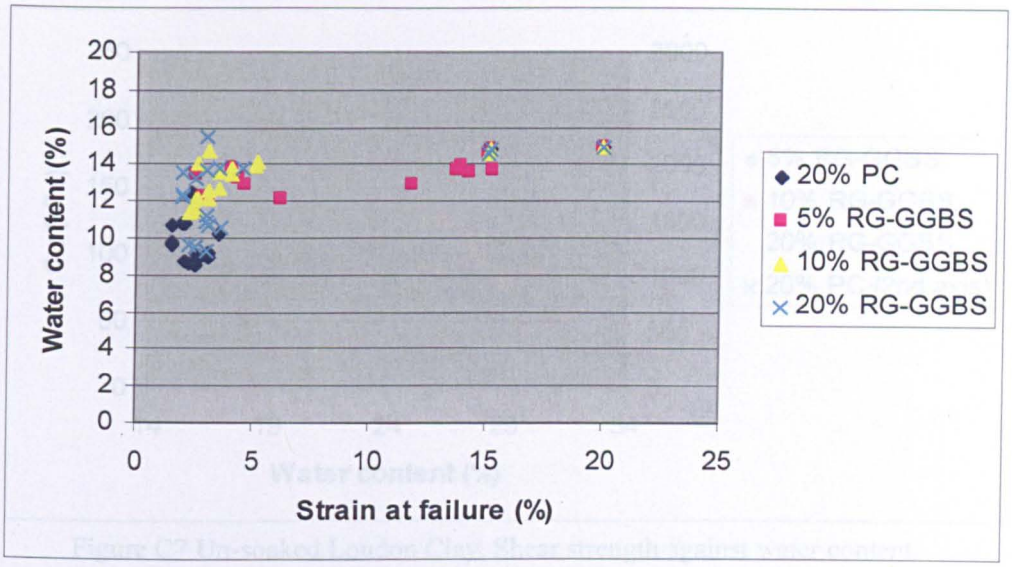



Figure C5 Un-soaked silty sand, water content against strain at failure.

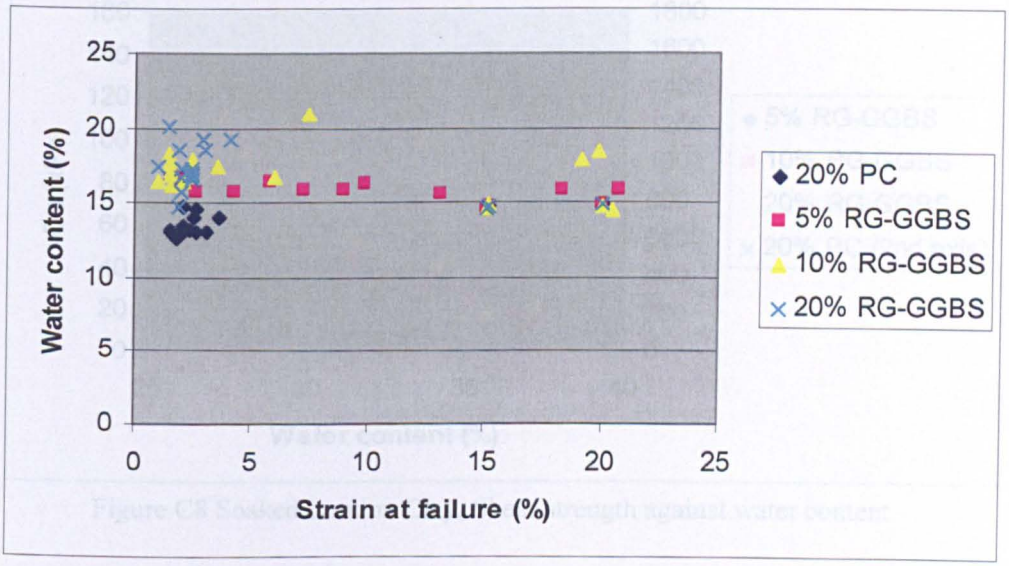



Figure C6 Soaked silty sand, water content against strain at failure.

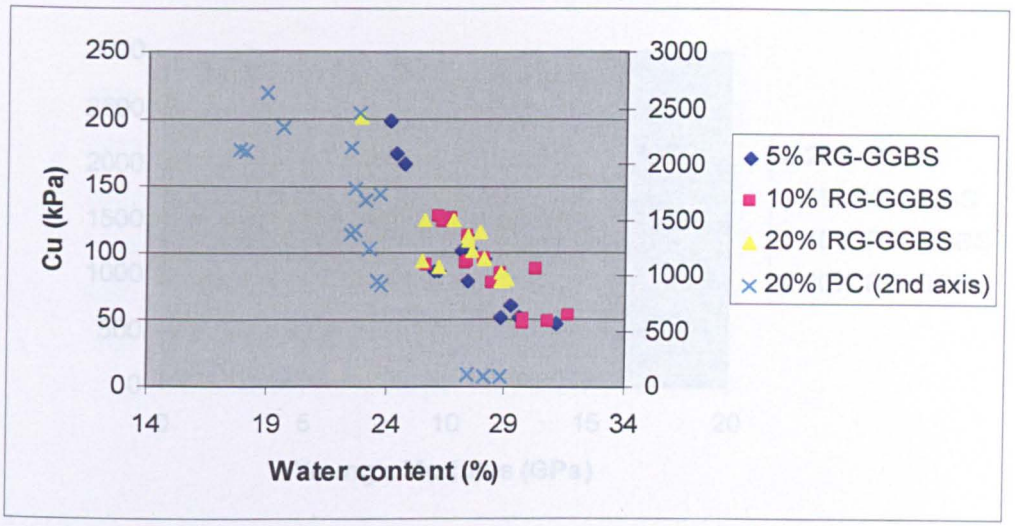



Figure C7 Un-soaked London Clay, Shear strength against water content.

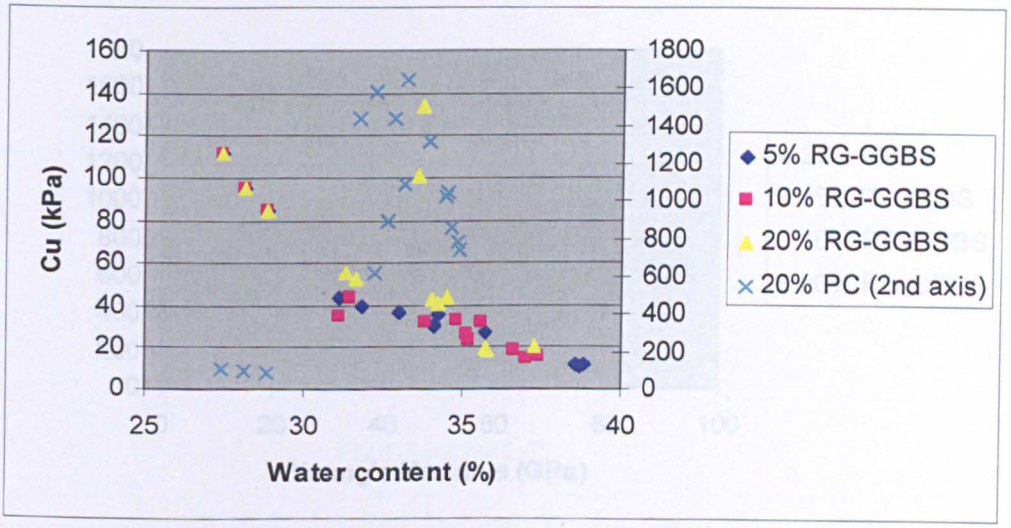



Figure C8 Soaked London Clay, Shear strength against water content.

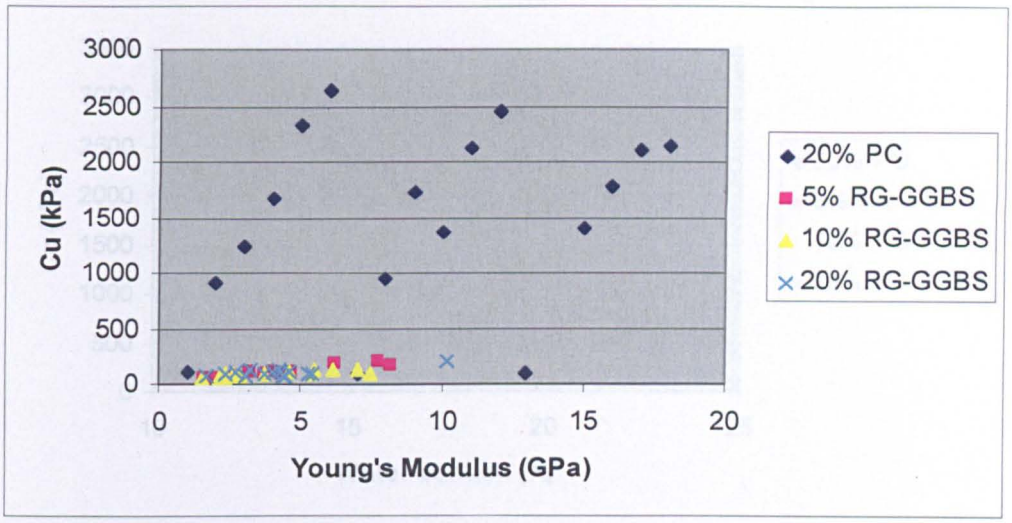



Figure C9 Un-soaked London Clay, Shear strength against Stiffness

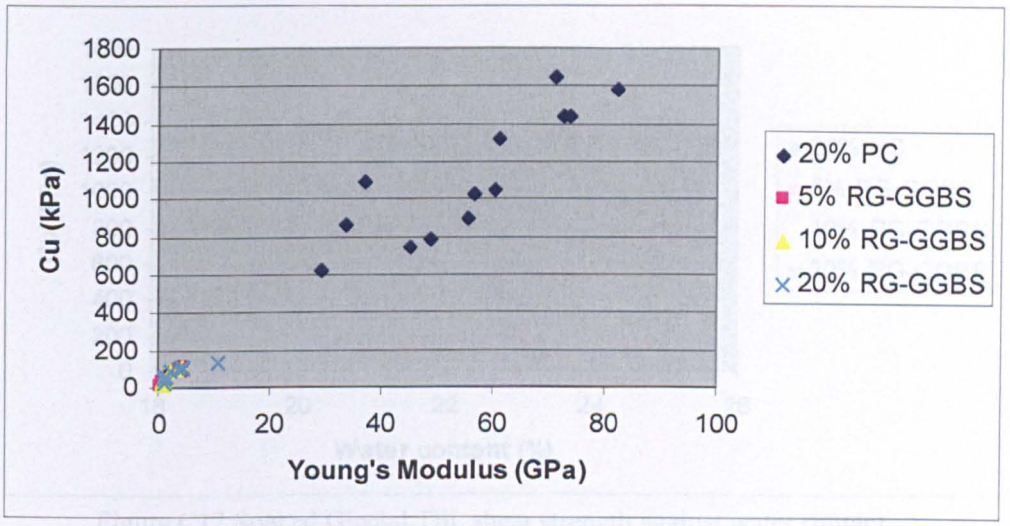



Figure C10 Soaked London Clay, Shear strength against Stiffness.

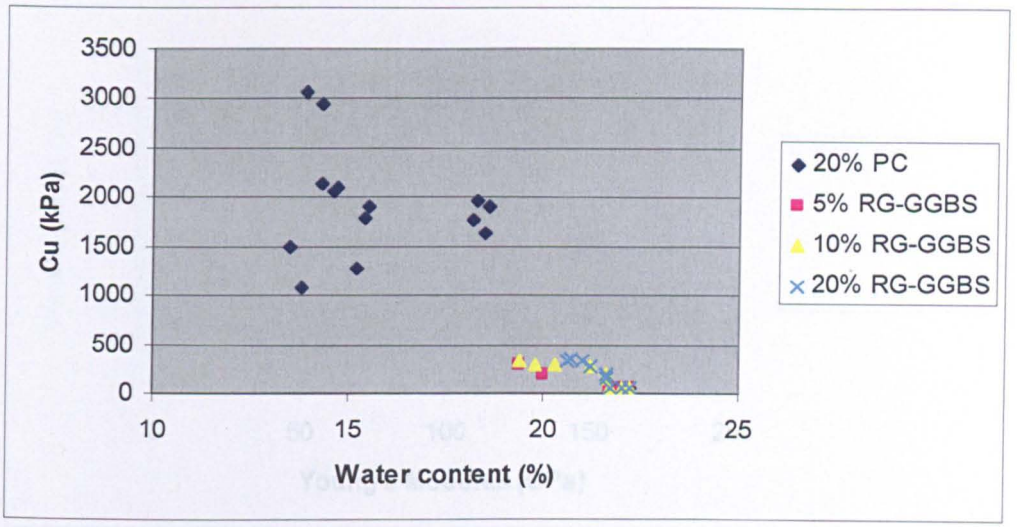



Figure C11 Un-soaked London Clay, Shear strength against water content.

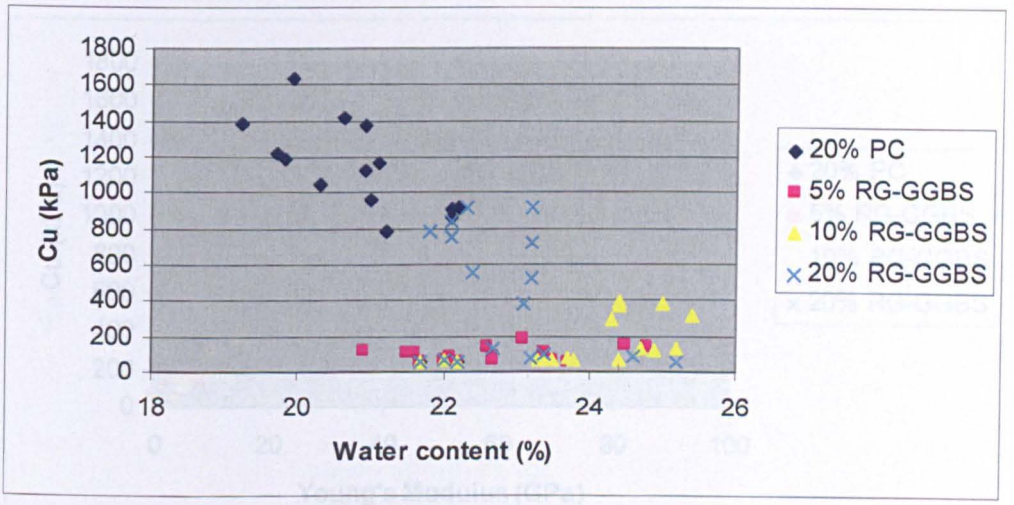



Figure C12 Soaked Glacial Till, shear strength against water content.

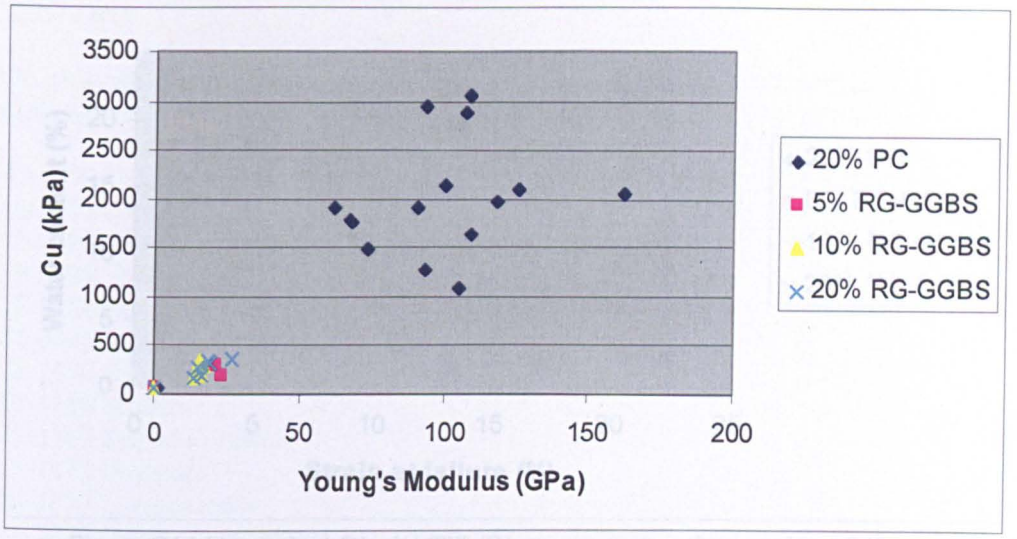



Figure C13 Un-soaked Glacial Till, shear strength against stiffness.

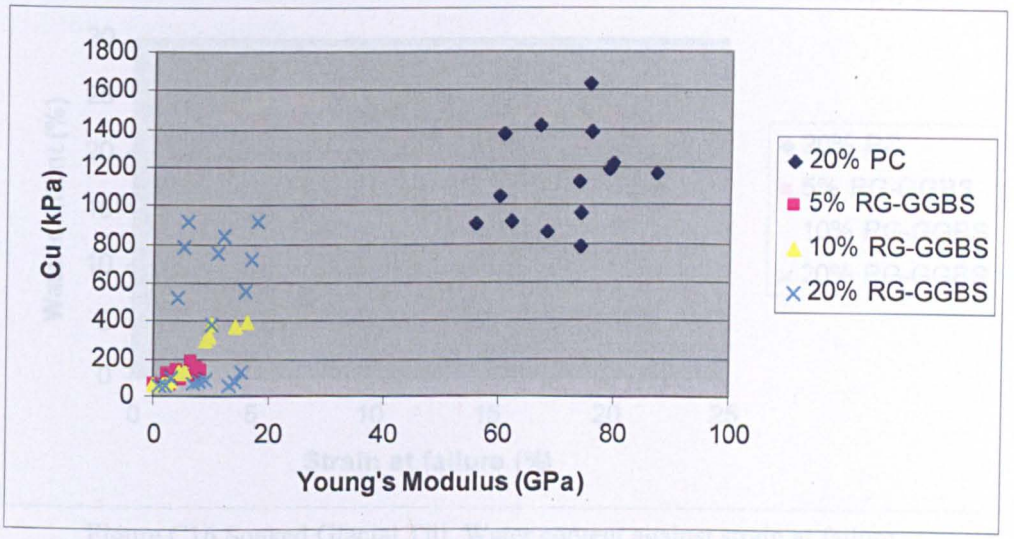



Figure C14 Soaked Glacial Till, shaer strength against stiffness.

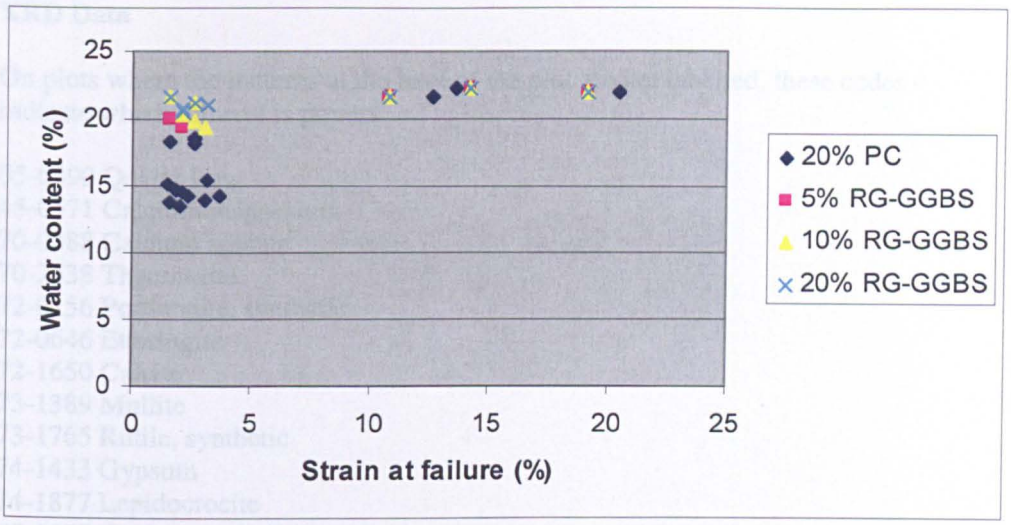



Figure C15 Un-soaked Glacial Till, Water content against strain at failure.

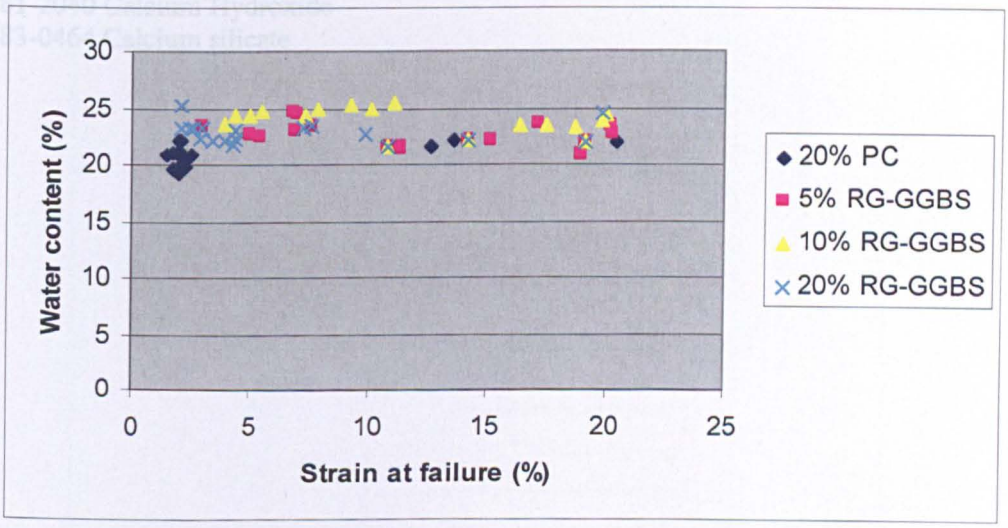
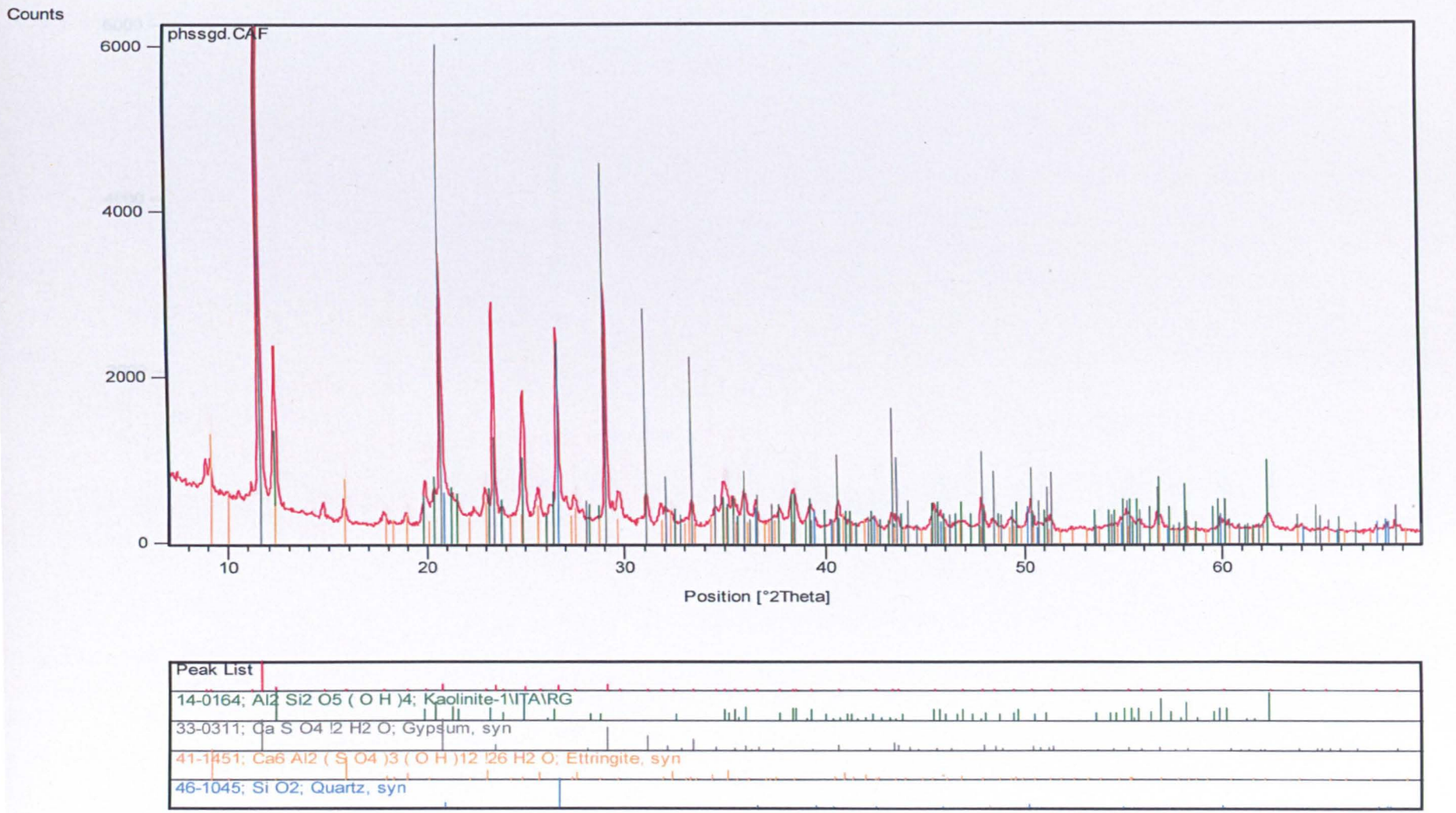



Figure C16 Soaked Glacial Till, Water content against strain at failure.

**XRD Data**

On plots where the patterns at the base of the plot are not labelled, these codes indicate which mineral is present.

05-0490 Quartz Low
45-0571 Calcium magnesium
70-0388 Calcium silicate
70-2438 Thaumasite
72-0156 Portlandite, synthetic
72-0646 Etteringite
72-1650 Calcite
73-1389 Mullite
73-1765 Rutile, synthetic
74-1433 Gypsum
74-1877 Lepidocrocite
75-1677 Gehlenite, synthetic
76-0841 Akermanite
79-1910 Quartz
81-2040 Calcium Hydroxide
83-0464 Calcium silicate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Position [°2Theta]
Peak List
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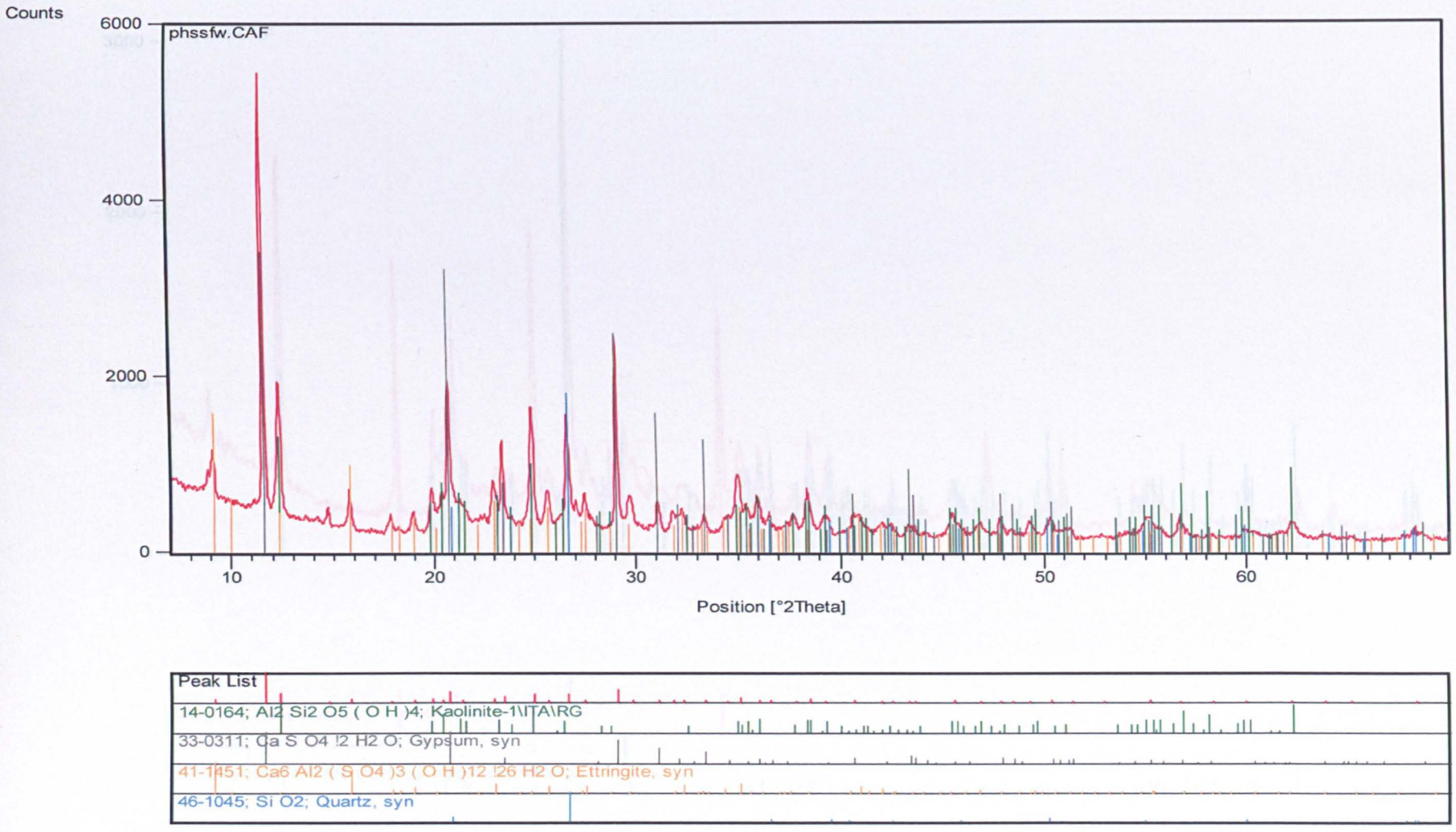
33-0311; Ca S O4 !2 H2 O; Gypsum, syn
41-1451; Ca6 Al2 ( S O4 )3 ( O H )12 !26 H2 O; Ettringite, syn
46-1045; Si O2; Quartz, syn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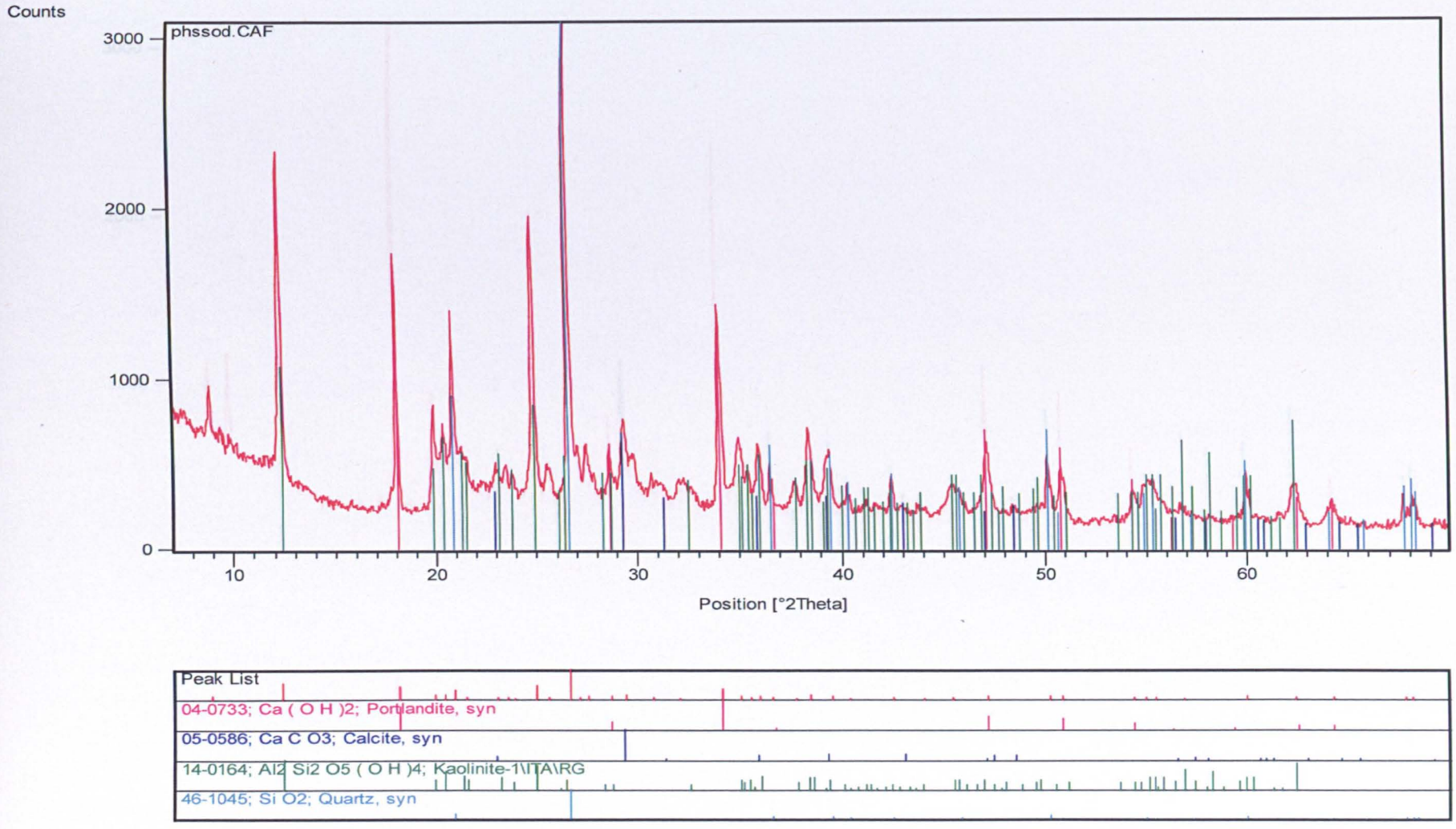
46-1045; Si O2; Quartz, syn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05-0586; Ca C O3; Calcite, syn
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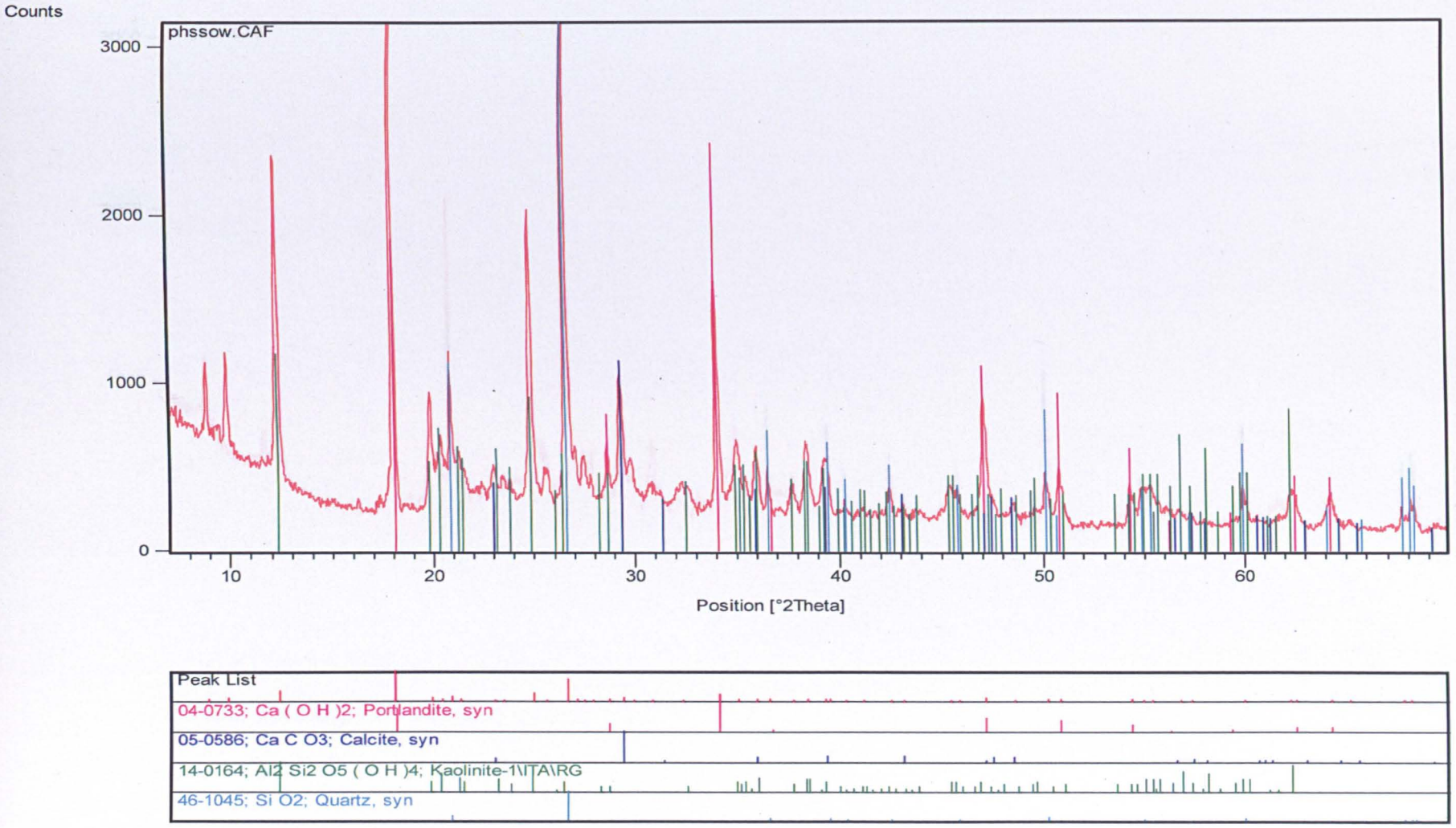
46-1045; Si O2; Quartz, syn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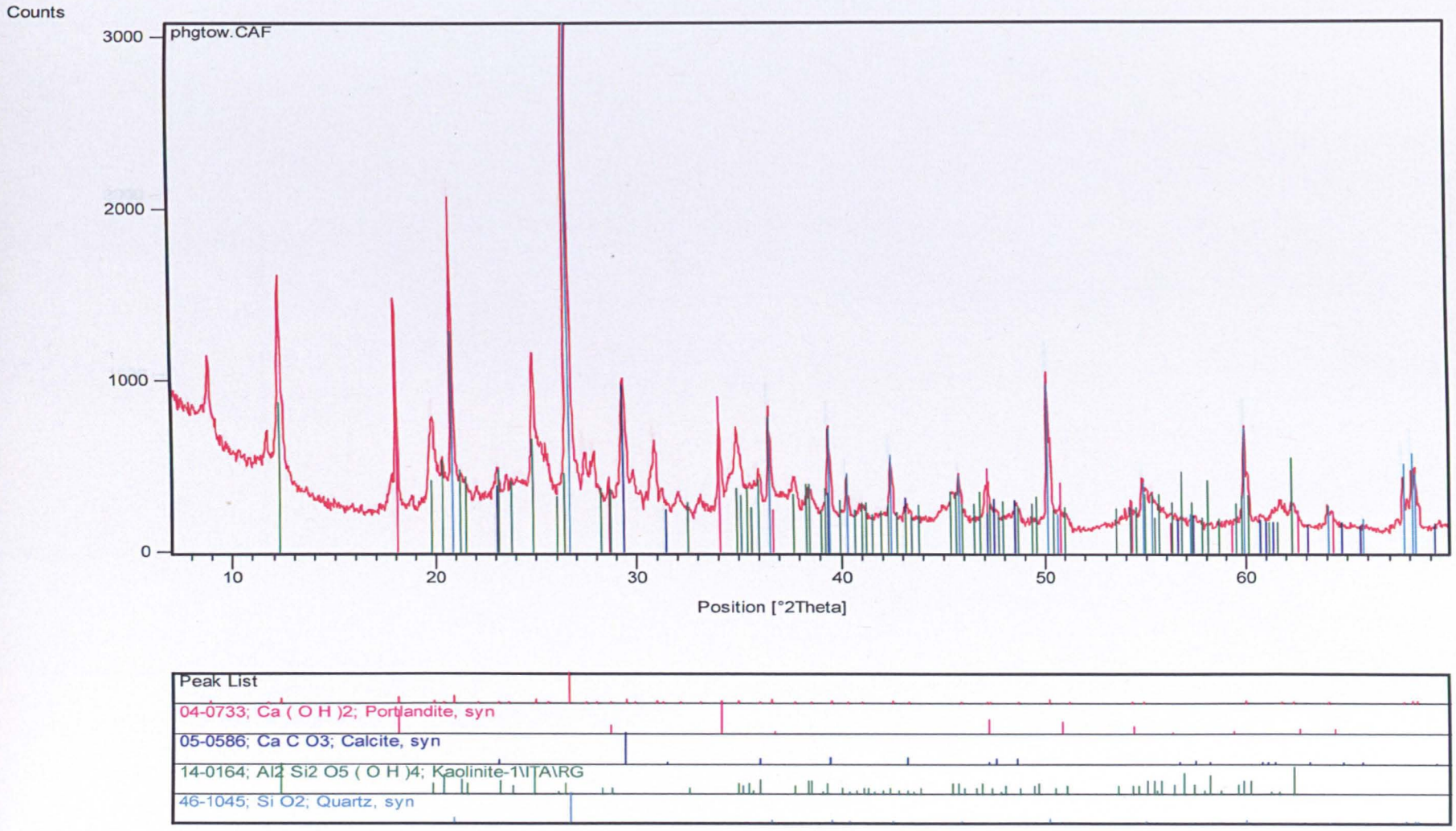
46-1045; Si O2; Quartz, syn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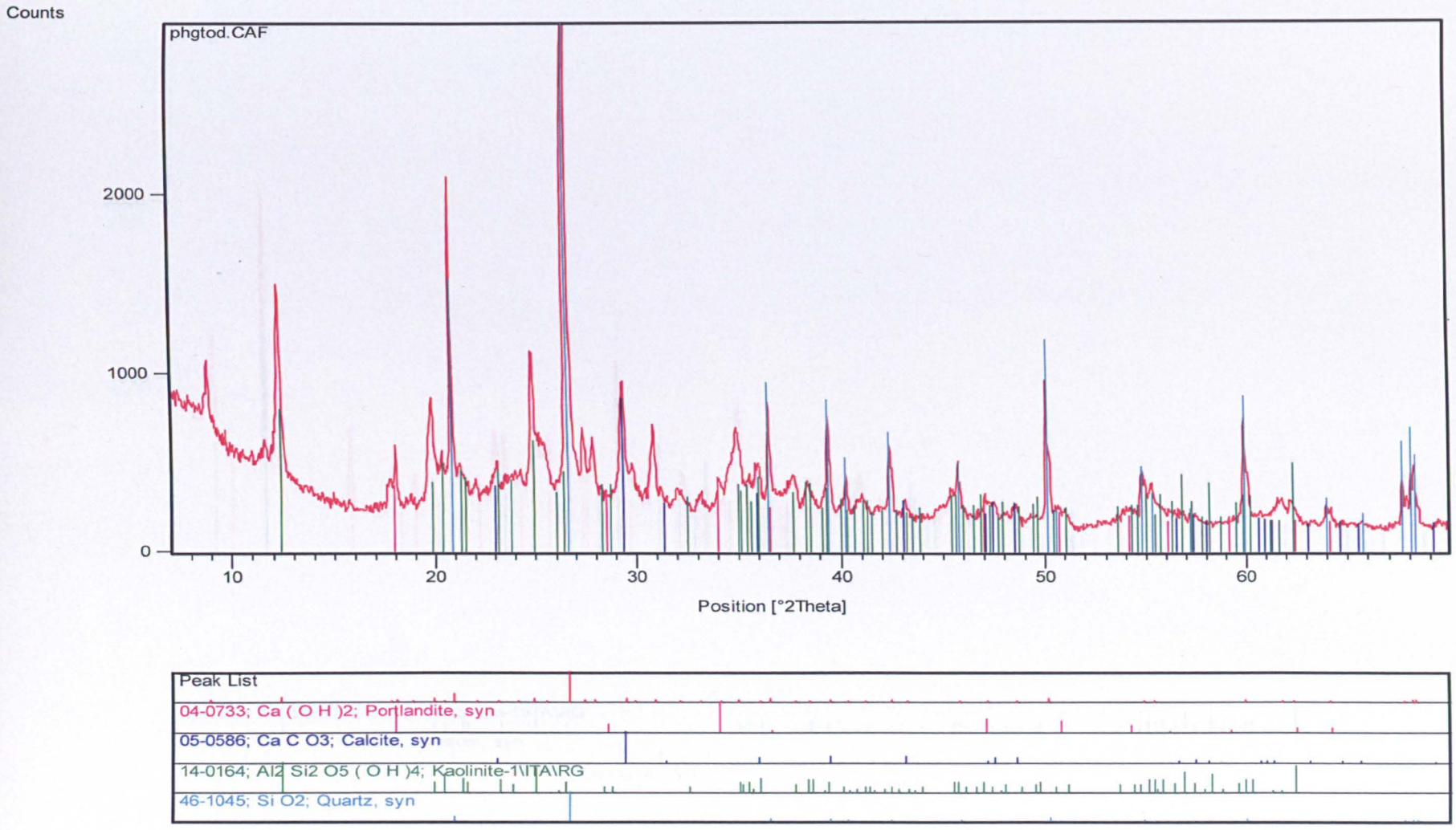
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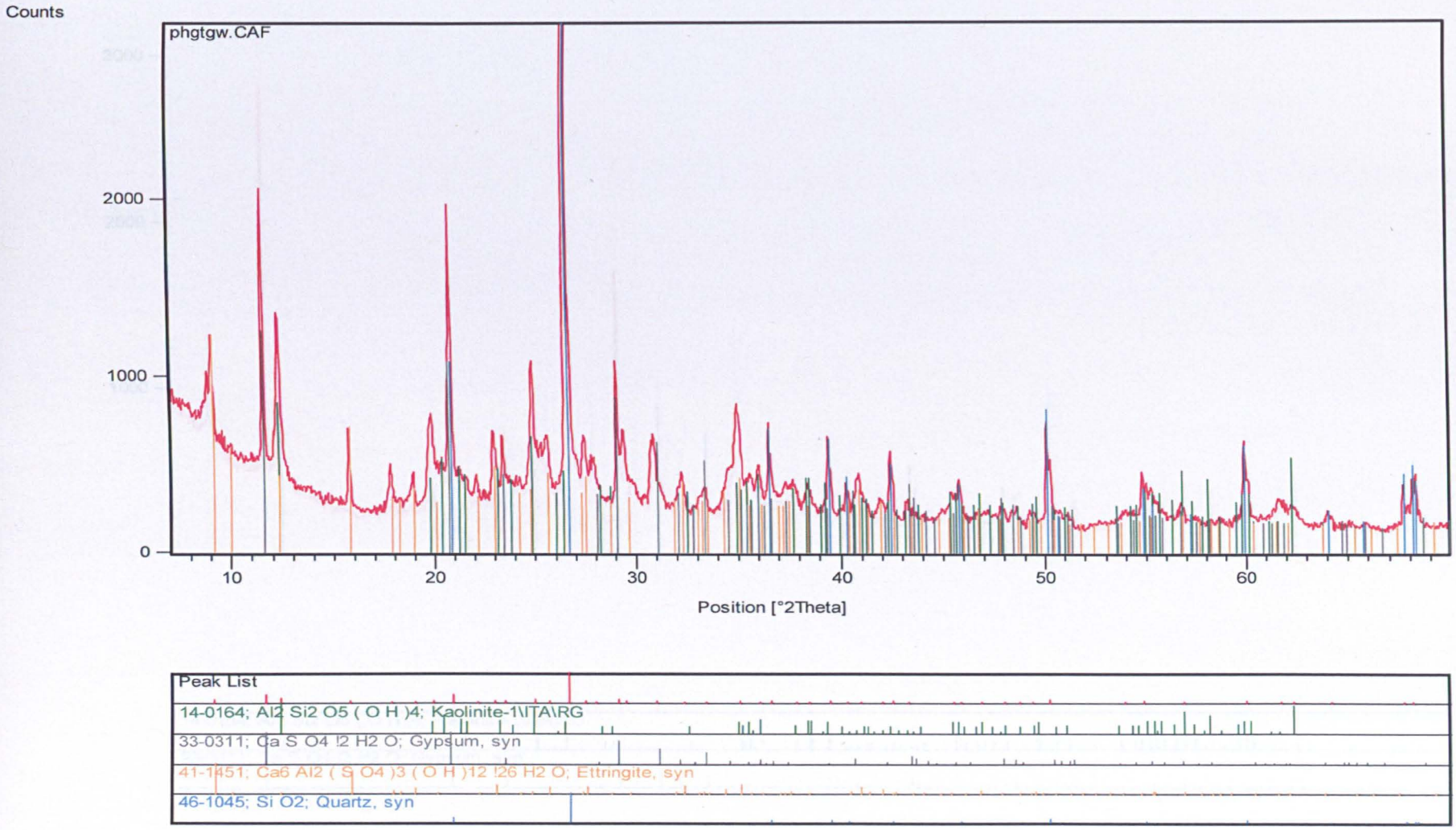
46-1045; Si O2; Quartz, syn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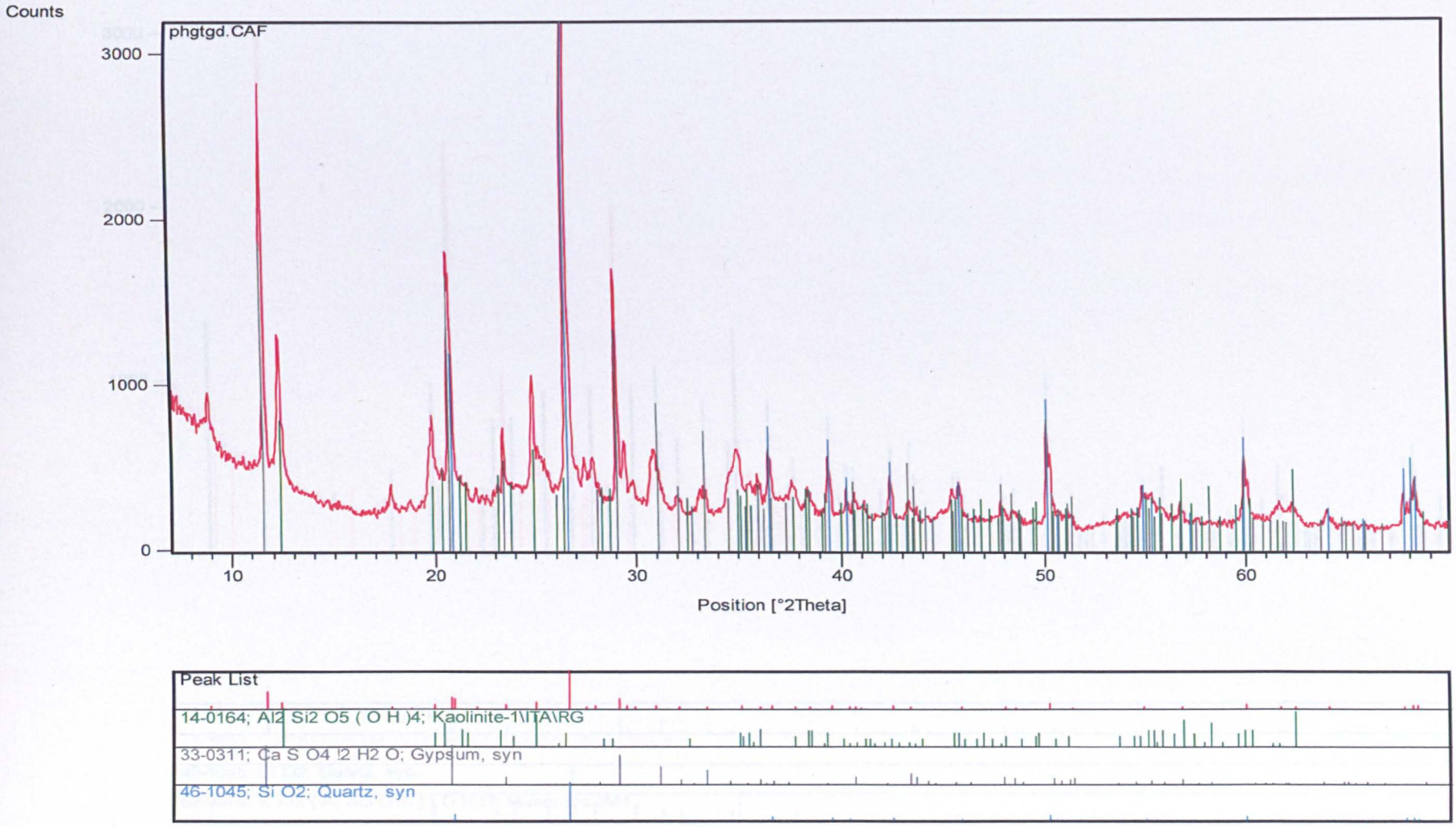
41-1451; Ca6 Al2 ( S O4 )3 ( O H )12 !26 H2 O; Ettringite, syn
46-1045; Si O2; Quartz, syn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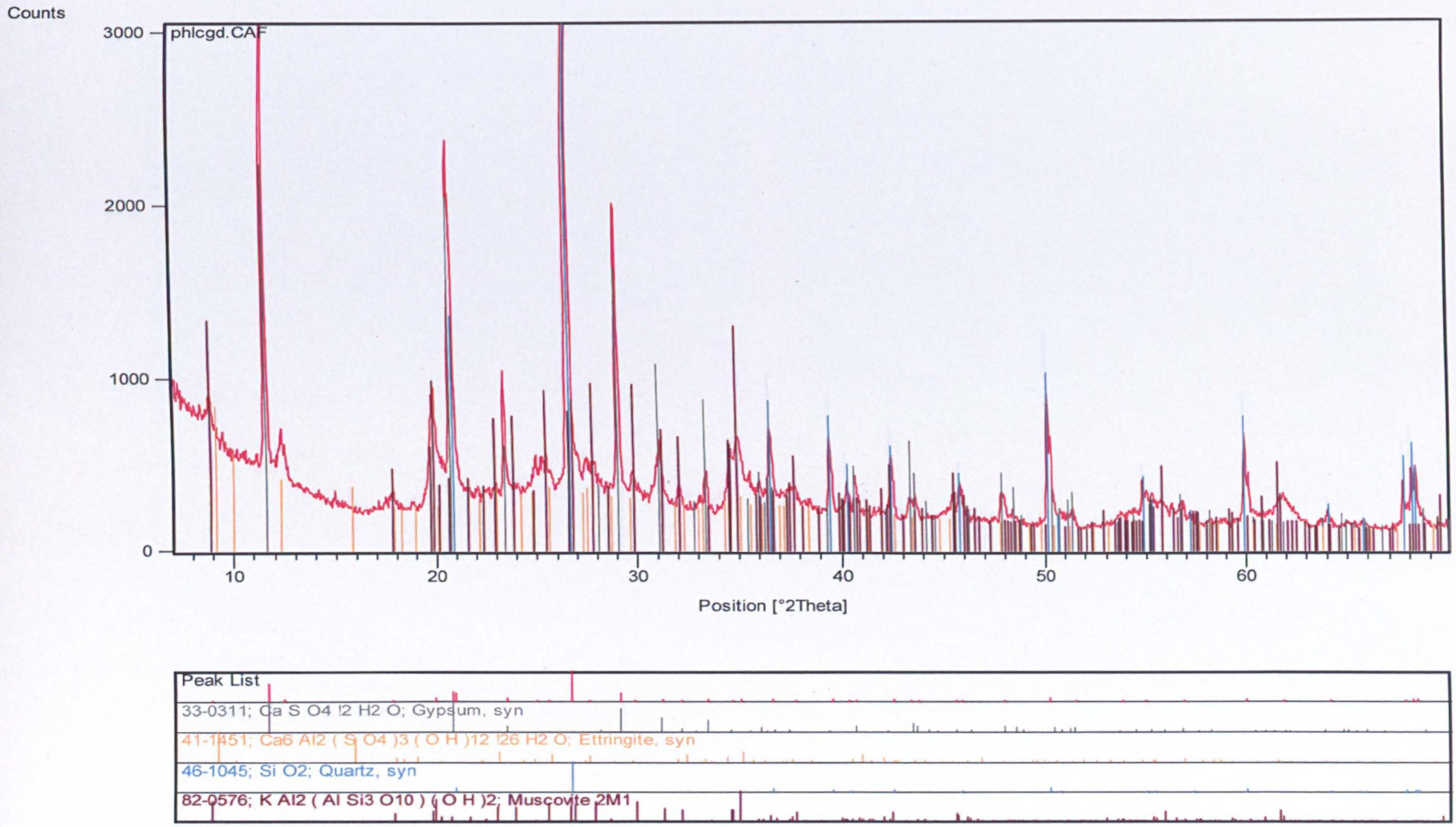
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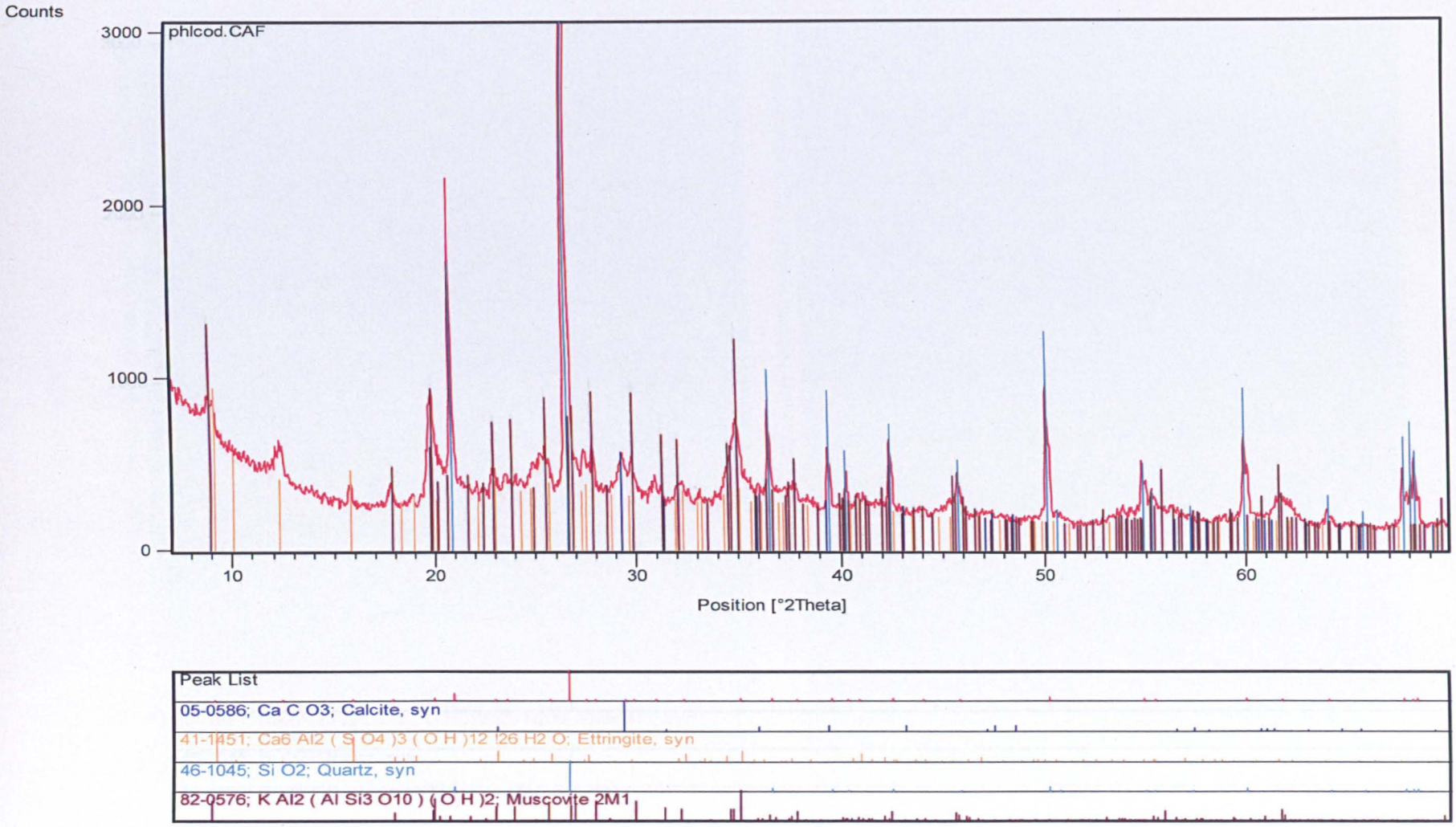
41-1451; Ca6 Al2 ( S O4 )3 ( O H )12 !26 H2 O; Ettringite, syn
46-1045; Si O2; Quartz, syn
82-0576; K Al2 ( Al Si3 O10 ) ( O H )2; Muscovite 2M1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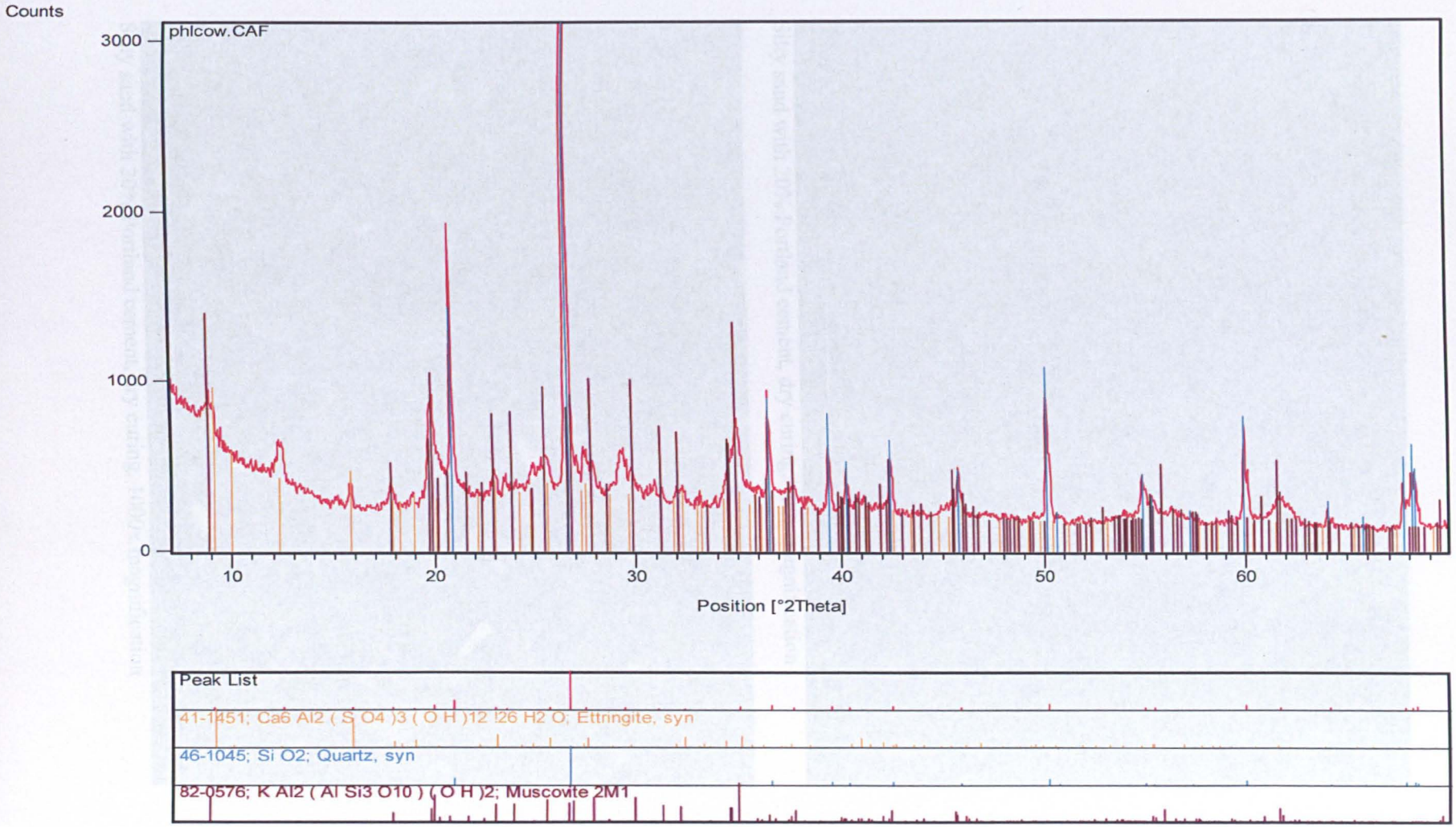
46-1045; Si O2; Quartz, syn
82-0576; K Al2 ( Al Si3 O10 ) ( O H )2; Muscovite 2M1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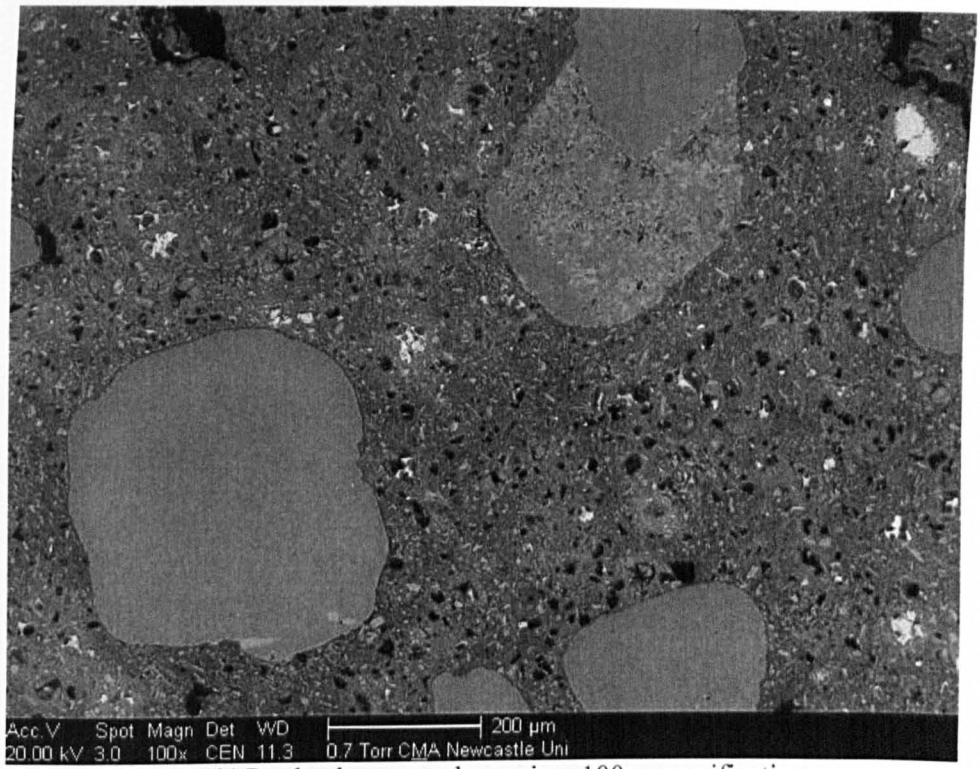
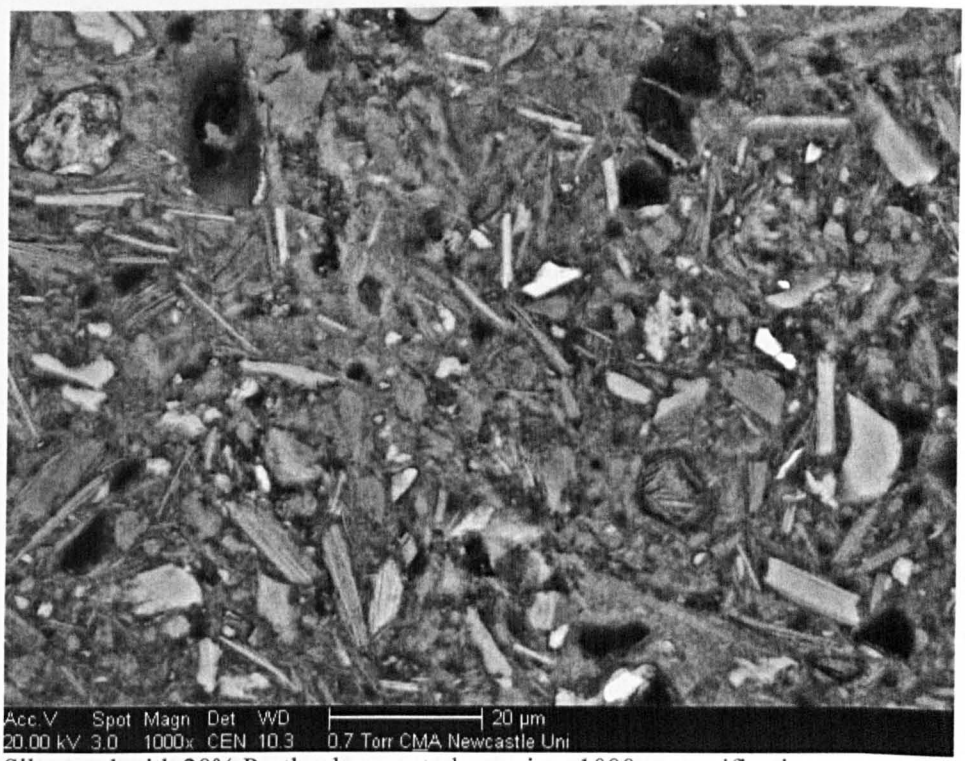
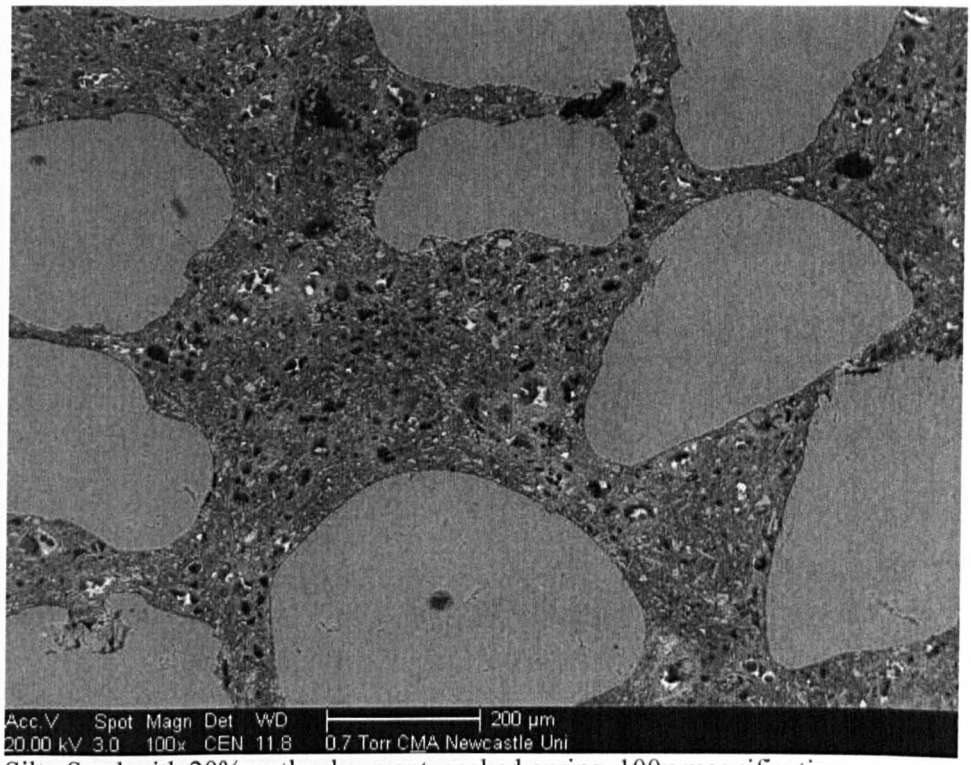


Silty sand with 20% Portland cement, dry curing, 100x magnification

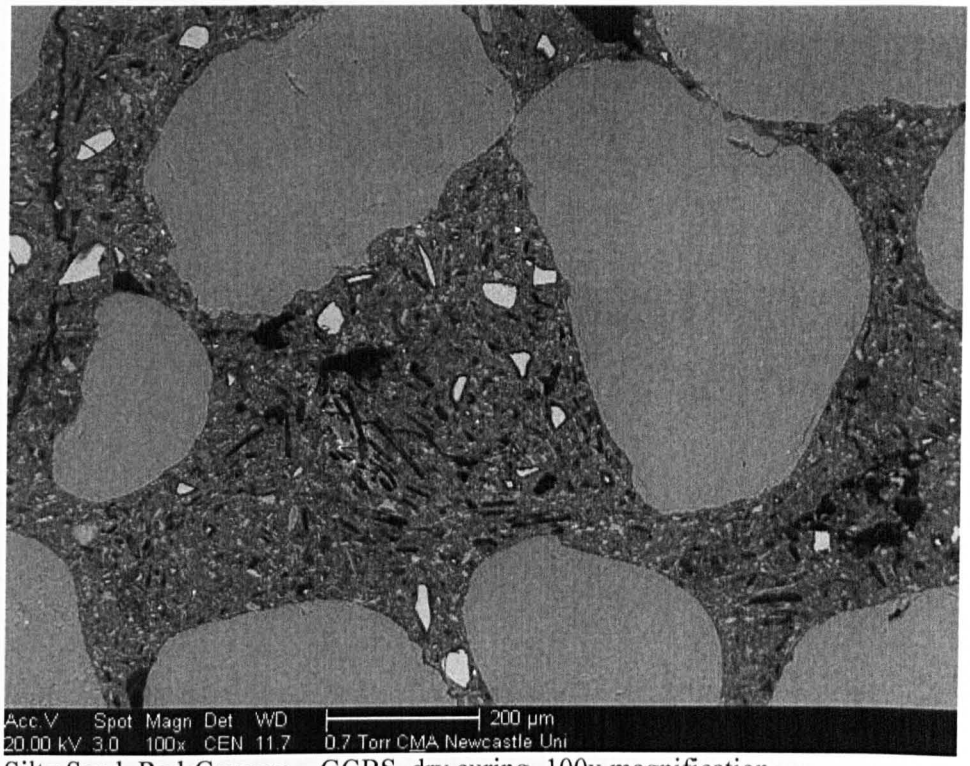


Silty sand with 20% Portland cement, dry curing, 1000x magnification

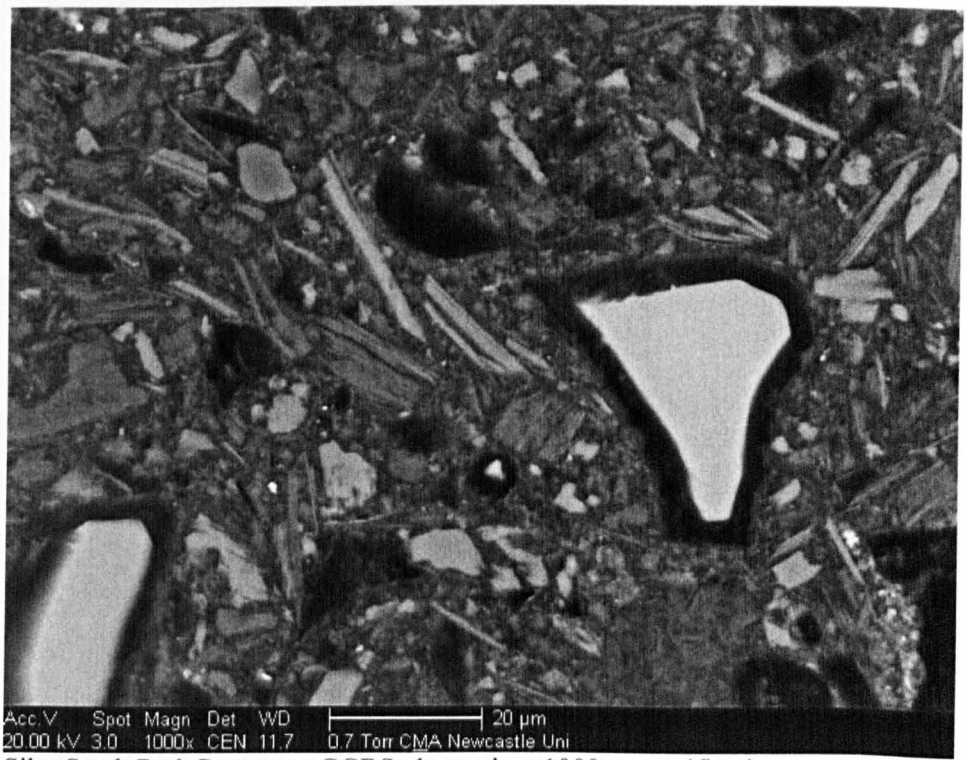


Silty Sand with 20%portland cement, soaked curing, 100x magnification

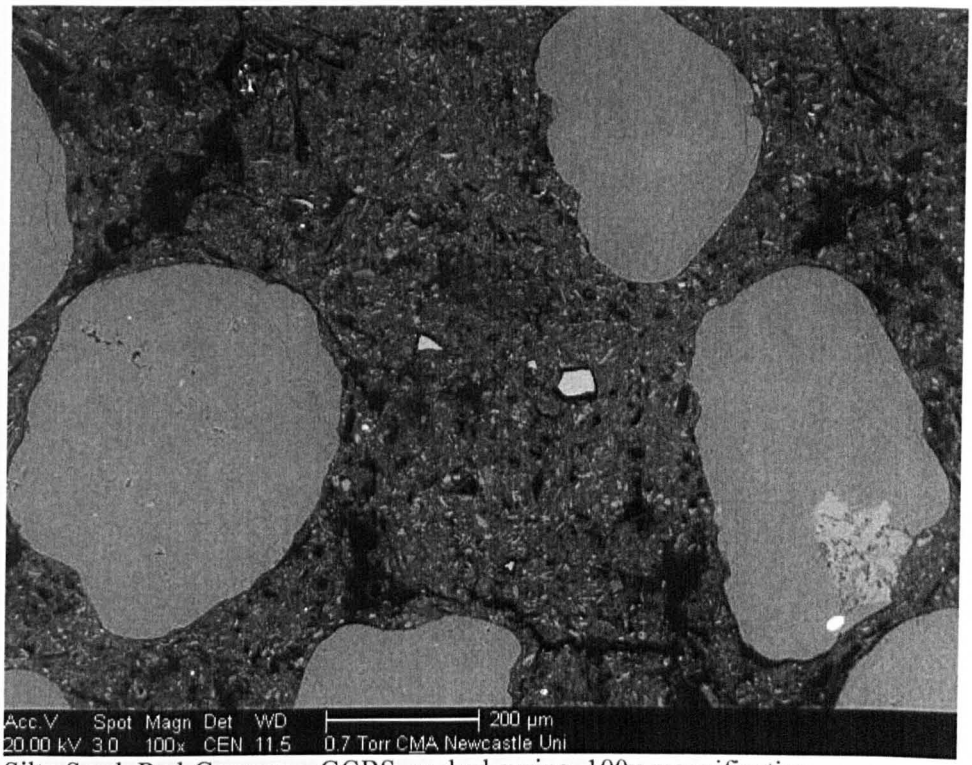
Silty Sand with 20%, portland cement, soaked curing, 1000x magnification



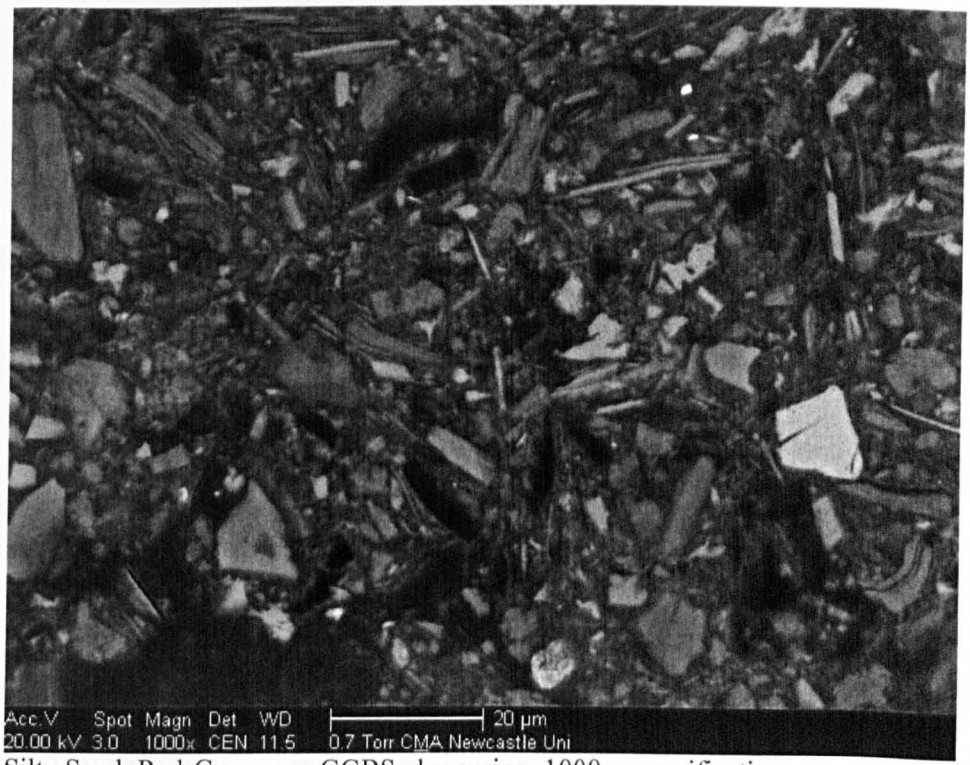
Silty Sand, Red-Gypsum – GGBS, dry curing, 100x magnification



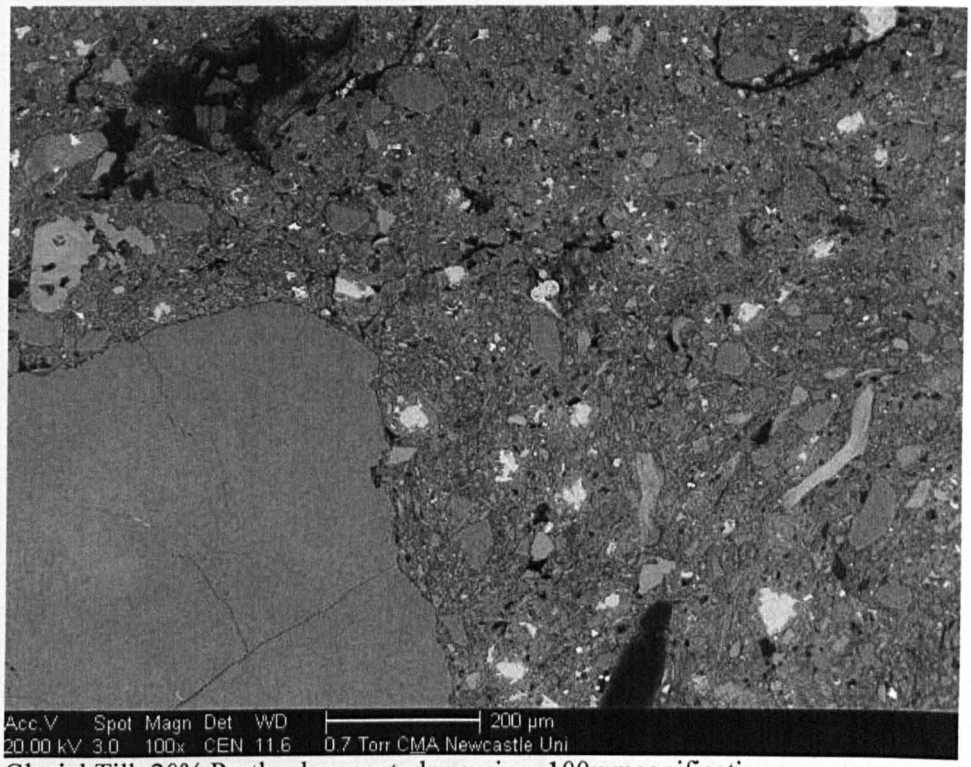
Silty Sand, Red-Gypsum – GGBS, dry curing, 1000x magnification



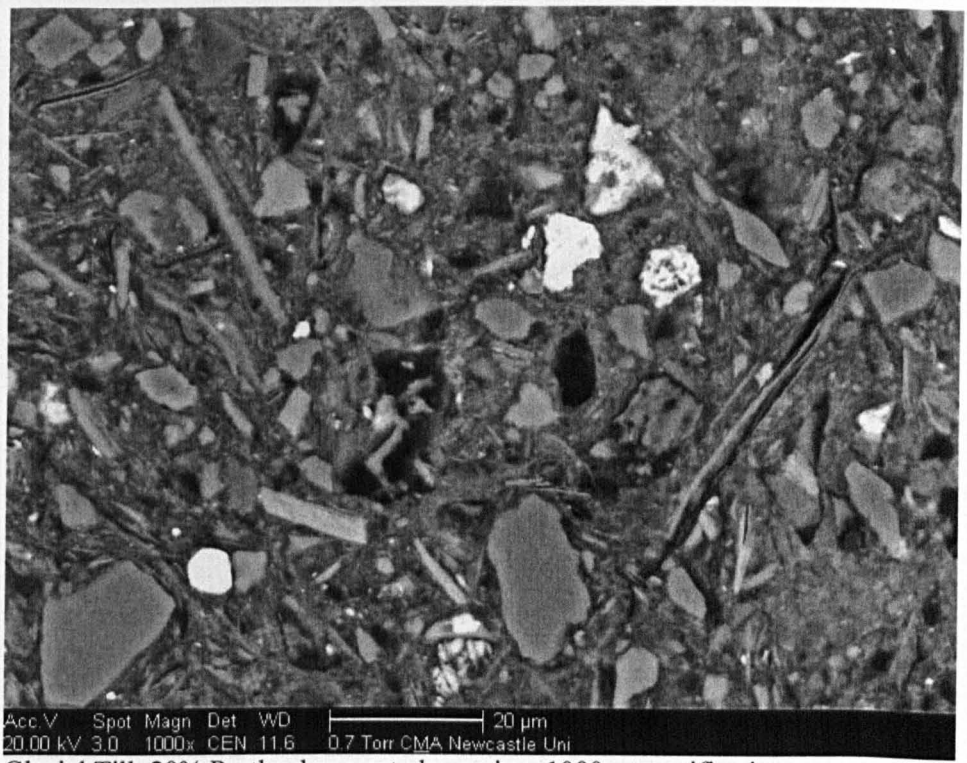
Silty Sand, Red-Gypsum – GGBS, soaked curing, 100x magnification



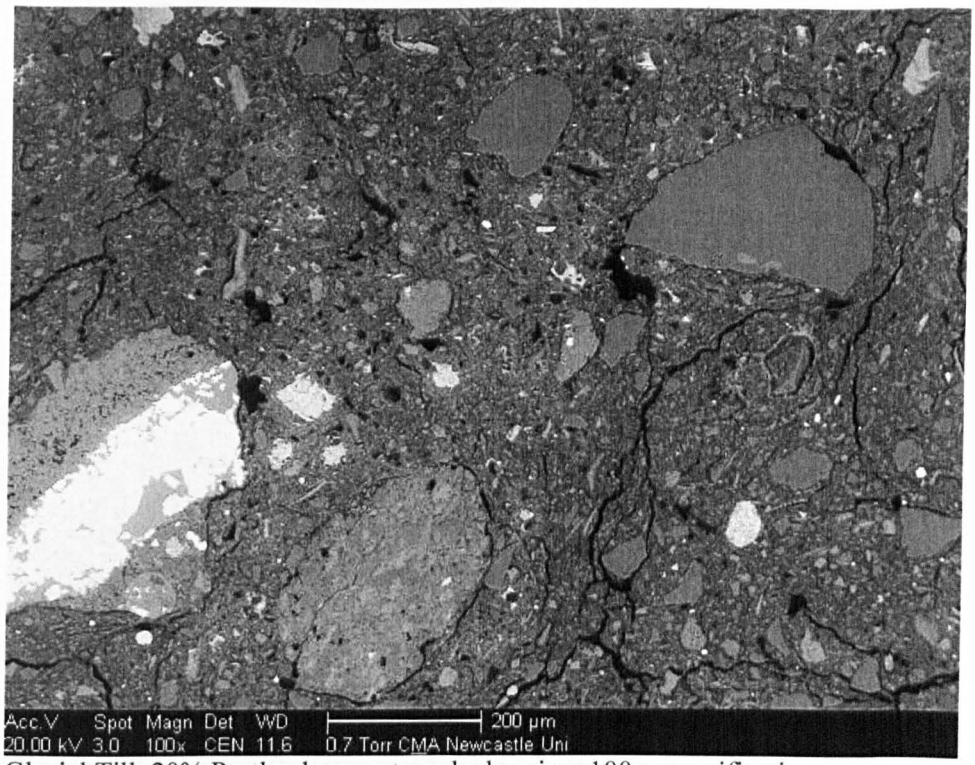
Silty Sand, Red-Gypsum – GGBS, dry curing, 1000x magnification



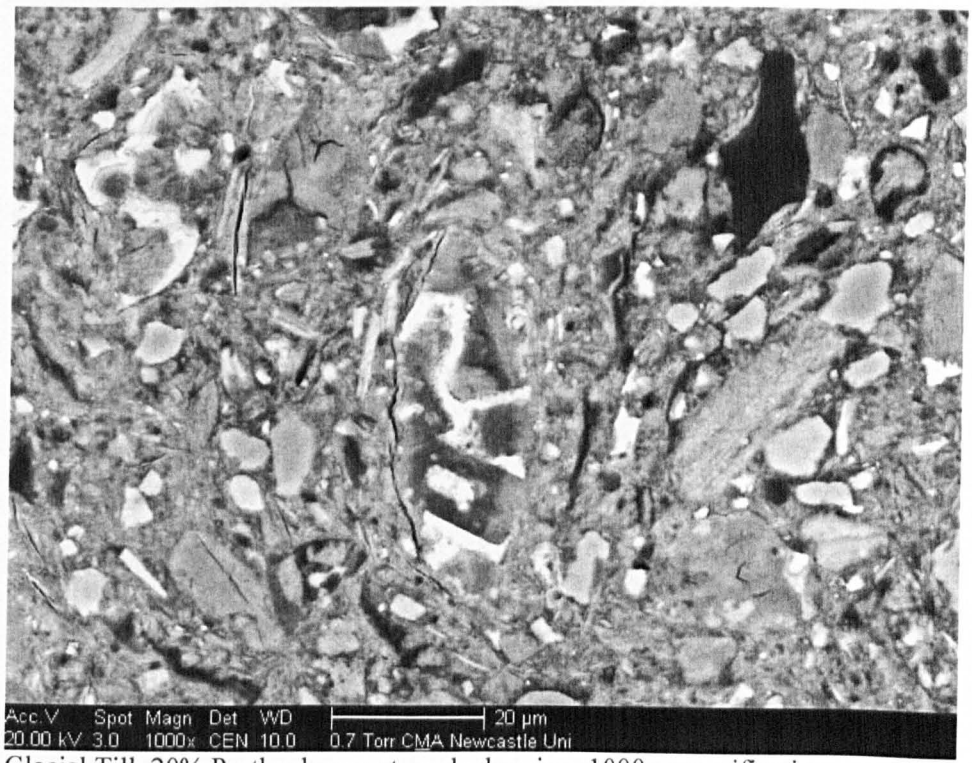
Glacial Till, 20% Portland cement, dry curing, 100x magnification



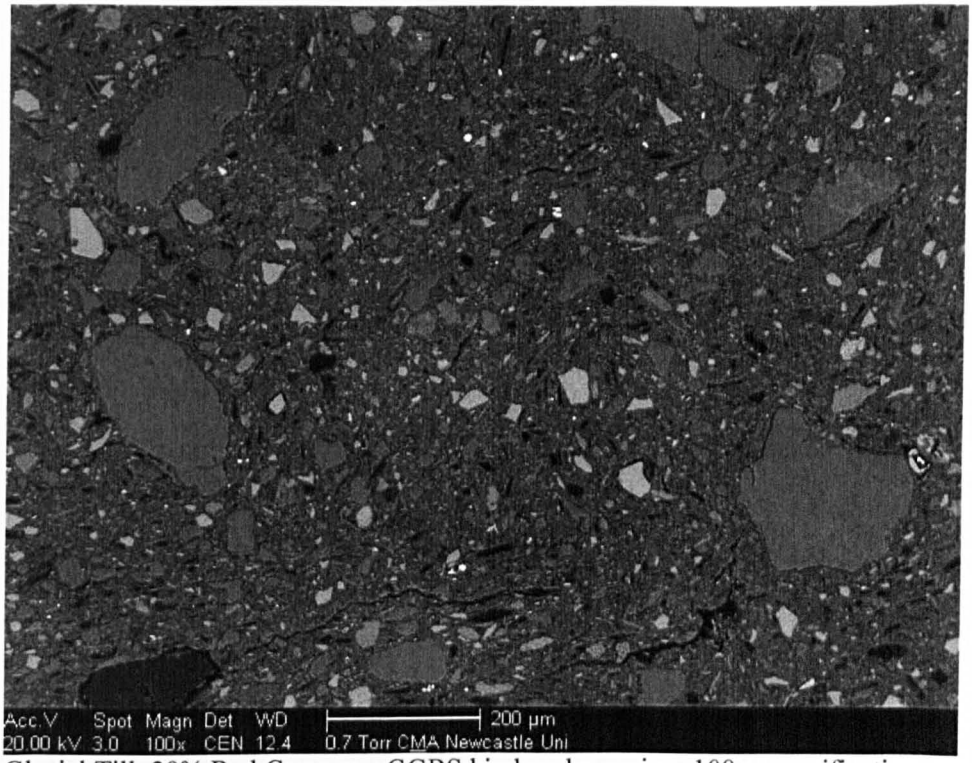
Glacial Till, 20% Portland cement, dry curing, 1000x magnification



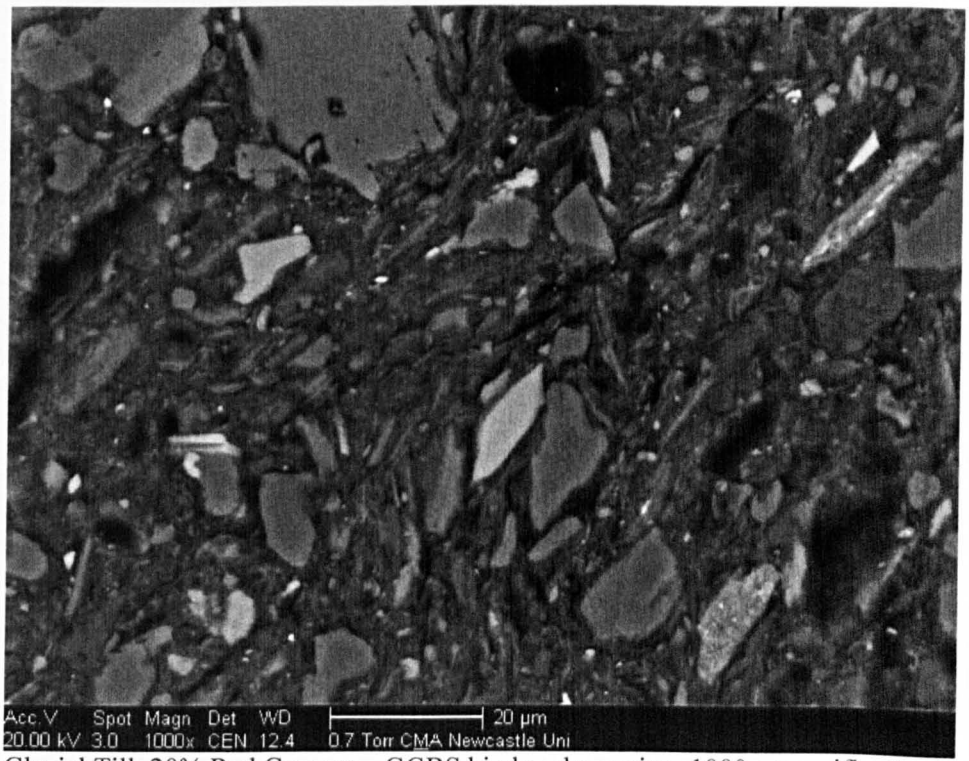
Glacial Till, 20% Portland cement, soaked curing, 100x magnification



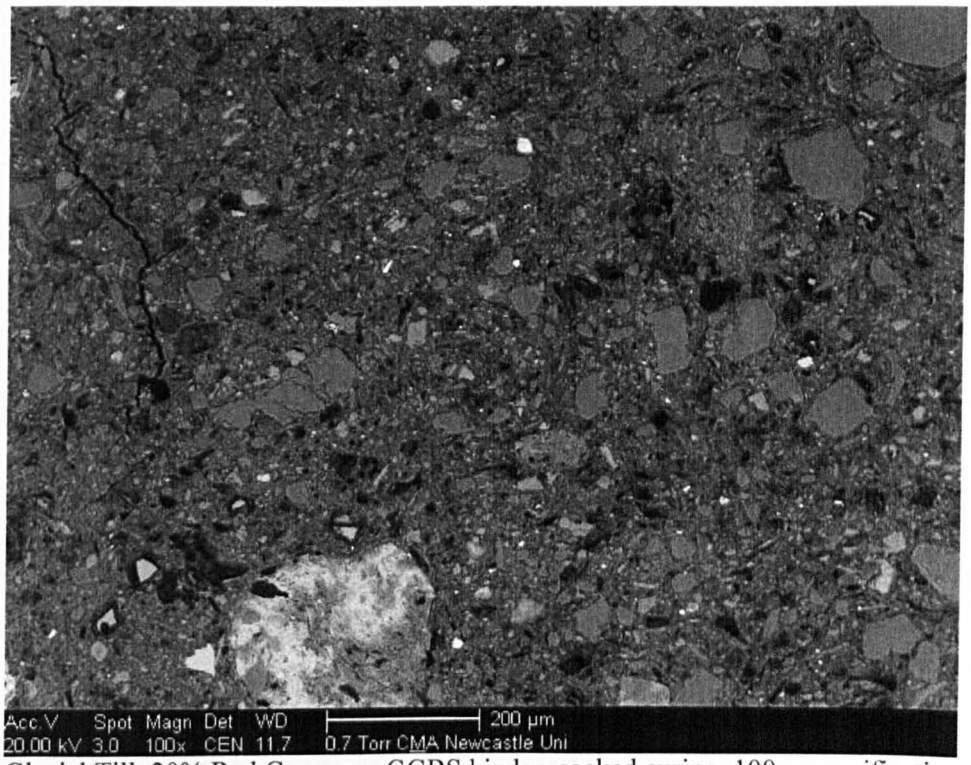
Glacial Till, 20% Portland cement, soaked curing, 1000x magnification



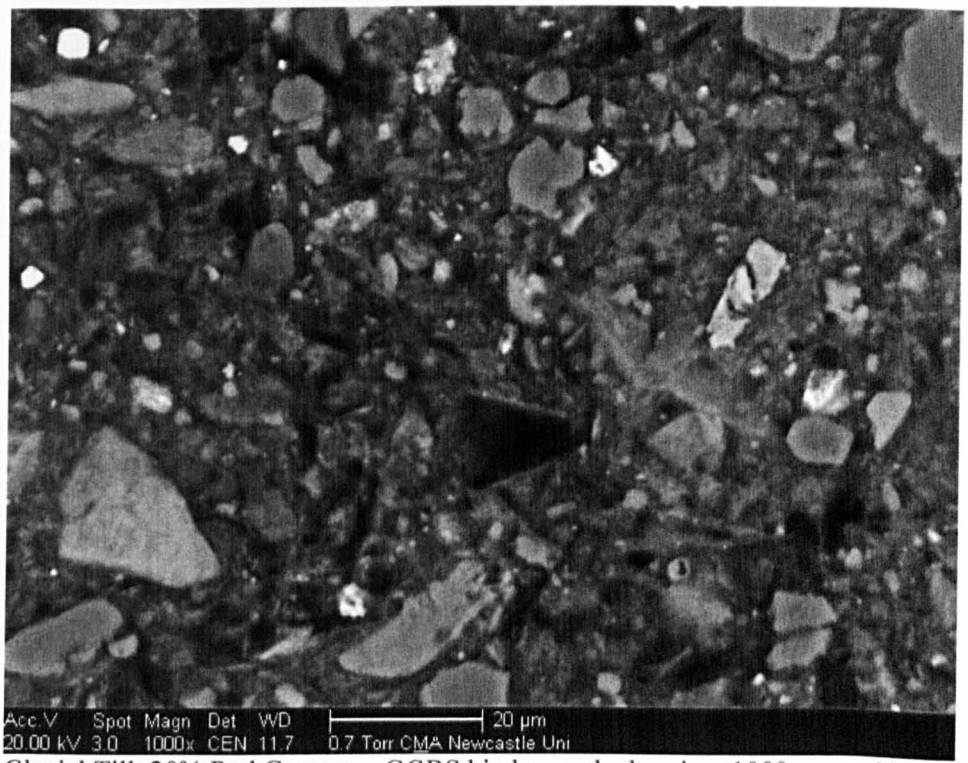
Glacial Till, 20% Red Gypsum - GGBS binder, dry curing, 100x magnification



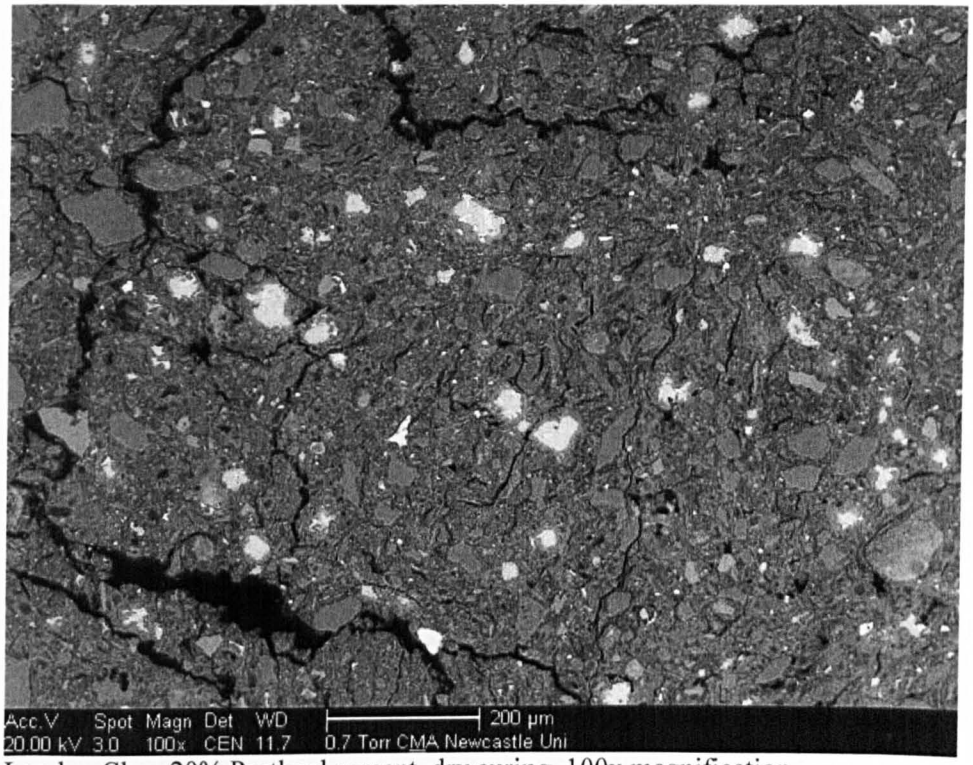
Glacial Till, 20% Red Gypsum - GGBS binder, dry curing, 1000x magnification



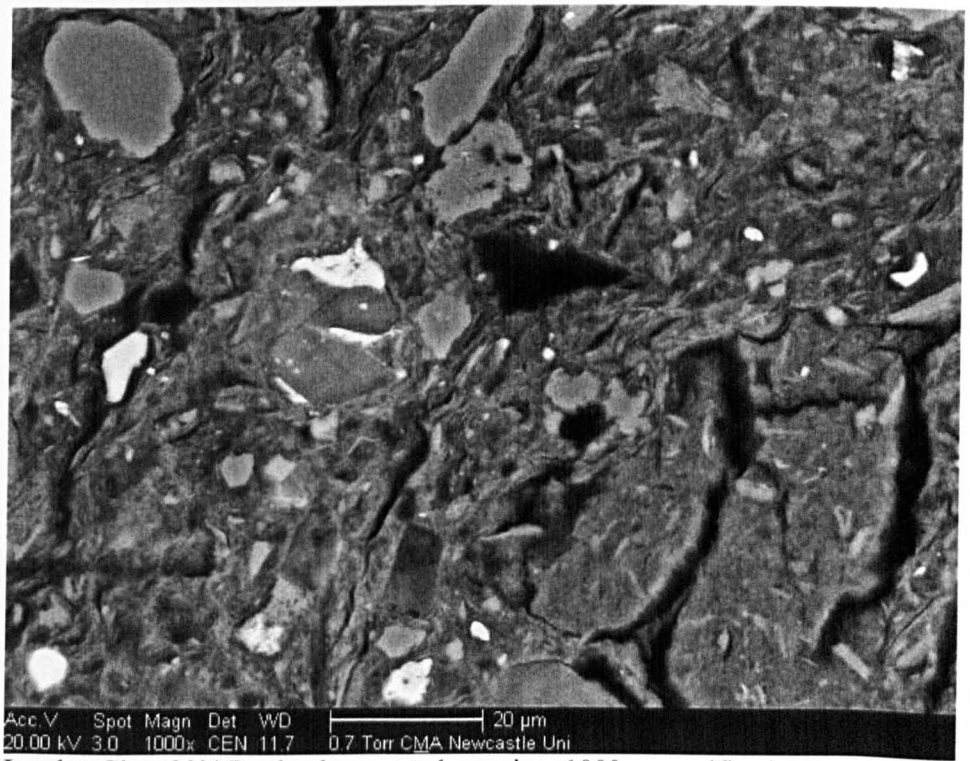
Glacial Till, 20% Red Gypsum - GGBS binder, soaked curing, 100x magnification



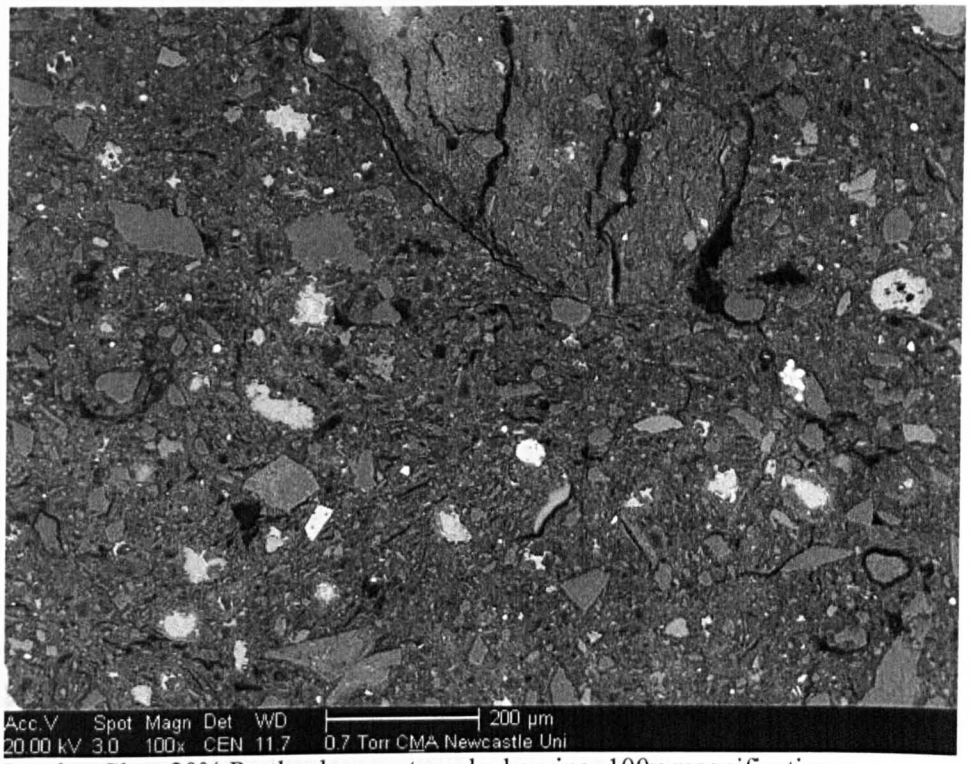
Glacial Till, 20% Red Gypsum - GGBS binder, soaked curing, 1000x magnification



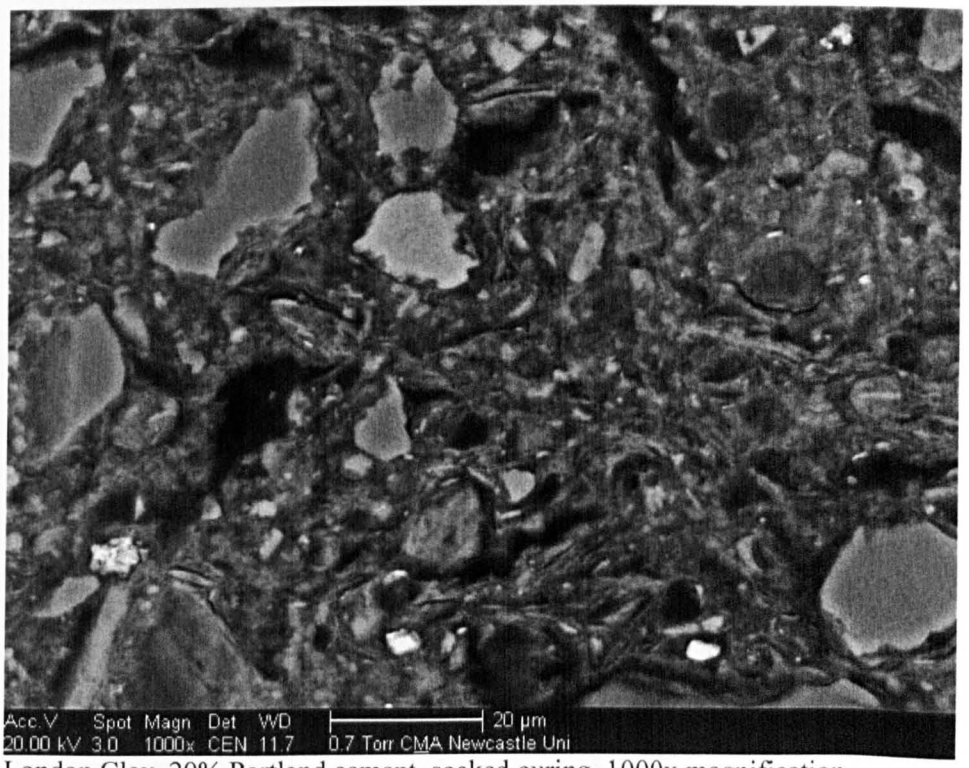
London Clay, 20% Portland cement, dry curing, 100x magnification



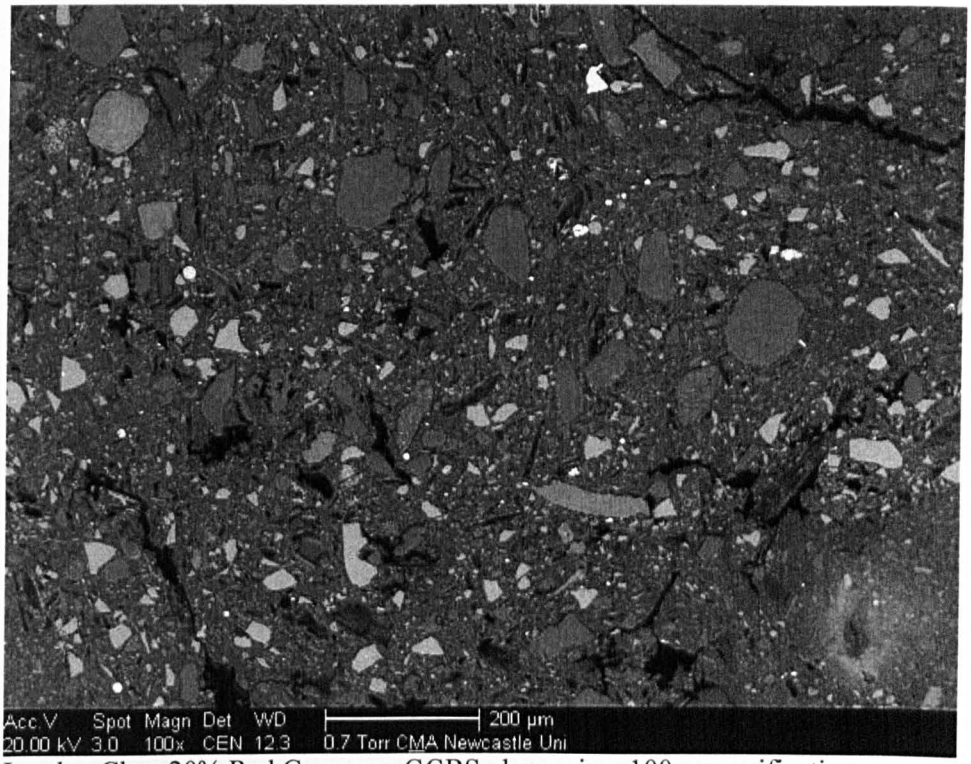
London Clay, 20% Portland cement, dry curing, 1000x magnification



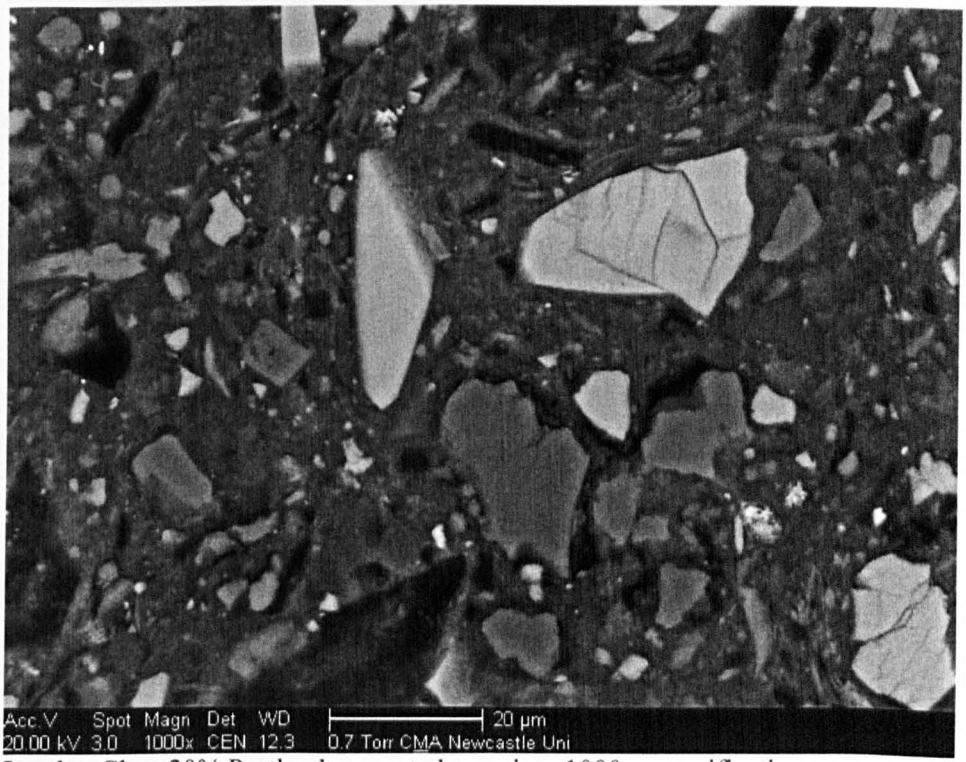
London Clay, 20% Portland cement, soaked curing, 100x magnification



London Clay, 20% Portland cement, soaked curing, 1000x magnification



London Clay, 20% Red Gypsum - GGBS, dry curing, 100x magnification



London Clay, 20% Portland cement, dry curing, 1000x magnification

RÖNTEC GmbH, Germany 25/08/2004

Quantax 1.2

Weight percent
Date: 25/08/2004

| Spectrum | Oxygen | Aluminium | Silicon | Magnesium | Calcium | Potassium | Sulfur | Iron |
|---|---|---|---|---|---|---|---|---|
| A1 Point0 | 52.09 | 5.11 | 7.97 | 0.52 | 19.44 | 0.80 | 0.52 | 13.54 |
| Point1 | 60.66 | 13.30 | 18.44 | | 6.53 | 1.06 | | |
| A1 Point2 | 56.03 | 12.64 | 20.47 | | 9.73 | 1.13 | | |
| Point3 | 52.42 | 9.32 | 26.25 | | 3.10 | 8.90 | | |
| A3 Point0 | 51.36 | 6.49 | 17.10 | 3.12 | 20.75 | 0.68 | 0.49 | |
| A3 Point1 | 58.25 | 11.51 | 18.91 | 1.10 | 4.71 | 2.09 | 1.26 | 2.17 |
| A3 Point3 | 58.08 | 11.06 | 19.87 | 1.33 | 6.94 | 1.23 | 1.48 | |
| A5 Point0 | 46.99 | 2.49 | 7.01 | | 5.14 | 0.79 | | 37.58 |
| A5 Point2 | 57.95 | 5.44 | 18.19 | 0.93 | 10.71 | 1.97 | | 4.81 |
| A5 Point3 | 55.58 | 8.09 | 18.34 | 1.39 | 9.14 | 2.88 | | 4.58 |
| Spectrum | Oxygen | Aluminium | Silicon | Magnesium | Calcium | Potassium | Sulfur | Iron |
| A6 Point0 | 58.60 | 7.58 | 14.45 | 4.94 | 3.64 | 2.01 | | 8.78 |
| A6 Point1 | 45.49 | 8.46 | 7.74 | 1.49 | 25.26 | | 0.76 | 10.81 |
| A7 Point0 | 51.05 | 6.34 | 16.47 | 2.75 | 19.56 | 0.98 | 0.44 | 2.41 |
| A7 Point1 | 52.51 | 6.88 | 13.20 | 2.39 | 1.90 | 1.23 | | 21.90 |
| A7 Point2 | 53.37 | 3.57 | 38.02 | 0.70 | 1.31 | 1.11 | | 1.92 |
| A8 Point0 | 55.84 | 10.64 | 19.63 | 1.74 | 3.29 | 3.46 | | 5.40 |
| A9 Point0 | 47.84 | 7.27 | 6.79 | 1.67 | 25.80 | 0.87 | 0.32 | 9.44 |
| A9Point1 | 58.18 | 6.51 | 17.28 | 3.41 | 5.41 | 2.87 | | 6.34 |
| A9 Point2 | 53.93 | 2.38 | 38.48 | 0.61 | 2.36 | 0.65 | | 1.58 |
| A11 Point0 | 51.10 | 6.80 | 17.93 | 3.01 | 17.36 | 1.12 | 0.58 | 2.10 |
| A11 Point1 | 55.96 | 2.30 | 38.90 | 0.70 | 0.32 | 0.01 | | 1.81 |
| A11 Point2 | 48.49 | 6.71 | 18.16 | 3.69 | 19.23 | 1.01 | 0.58 | 2.11 |
| A11 Point3 | 56.29 | 9.77 | 21.04 | 1.59 | 2.72 | 2.65 | 0.52 | 5.43 |

RÖNTEC GmbH, Germany 31/08/2004

Quantax 1.2

Weight percent
Date: 31/08/2004

| Spectrum | Oxygen | Magnesium | Aluminium | Silicon | Potassium | Calcium | Iron | Sulfur |
|---|---|---|---|---|---|---|---|---|
| A6 Point0 | 58.60 | 4.94 | 7.58 | 14.45 | 2.01 | 3.64 | 8.78 | |
| A6 Point1 | 45.49 | 1.49 | 8.46 | 7.74 | | 25.26 | 10.81 | 0.76 |
| A7 Point0 | 51.05 | 2.75 | 6.34 | 16.47 | 0.98 | 19.56 | 2.41 | 0.44 |
| A7 Point1 | 52.51 | 2.39 | 6.88 | 13.20 | 1.23 | 1.90 | 21.90 | |
| A7 Point2 | 53.37 | 0.70 | 3.57 | 38.02 | 1.11 | 1.31 | 1.92 | |
| A8 Point0 | 55.84 | 1.74 | 10.64 | 19.63 | 3.46 | 3.29 | 5.40 | |
| A9 Point0 | 47.84 | 1.67 | 7.27 | 6.79 | 0.87 | 25.80 | 9.44 | 0.32 |
| A9Point1 | 58.18 | 3.41 | 6.51 | 17.28 | 2.87 | 5.41 | 6.34 | |
| A9 Point2 | 53.93 | 0.61 | 2.38 | 38.48 | 0.65 | 2.36 | 1.58 | |
| A11 Point0 | 51.10 | 3.01 | 6.80 | 17.93 | 1.12 | 17.36 | 2.10 | 0.58 |
| A11 Point1 | 55.96 | 0.70 | 2.30 | 38.90 | 0.01 | 0.32 | 1.81 | |
| A11 Point2 | 48.49 | 3.69 | 6.71 | 18.16 | 1.01 | 19.23 | 2.11 | 0.58 |
| A11 Point3 | 56.29 | 1.59 | 9.77 | 21.04 | 2.65 | 2.72 | 5.43 | 0.52 |

# Appendix D

# Blockwork testing data

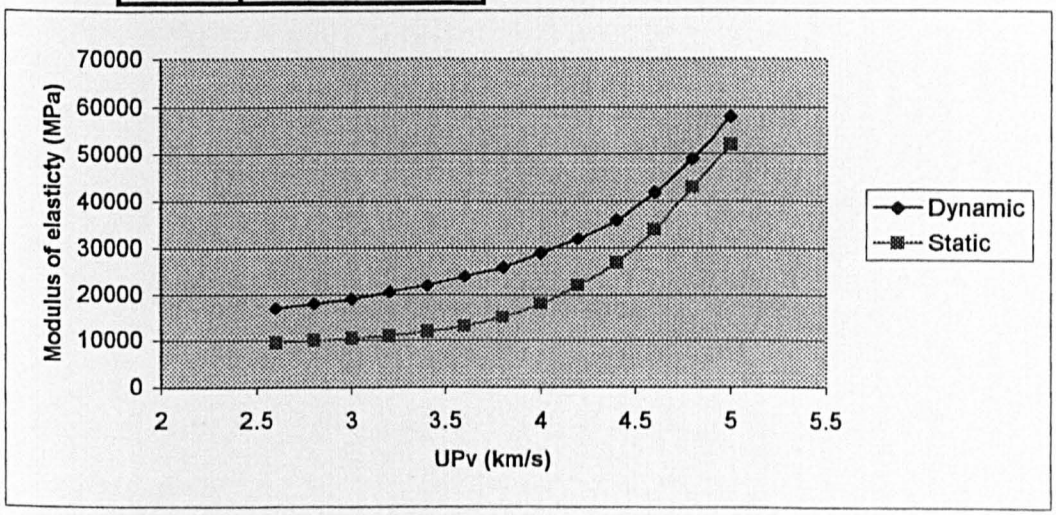
# Appendix D Contents

UPv – Dynamic Modulus of elasticity plot (adapted from BS 1881)

**Conversion from Upv to Modulus of elasticty used in Chapter 6, Modified from BS 1881**

| Upv (km/s) | dynamic Modulus of elasticty (Mpa) | static modulus of elastcity (Mpa) |
|---|---|---|
| 2.6 | 17000 | 9600 |
| 2.8 | 18000 | 10000 |
| 3 | 19000 | 10400 |
| 3.2 | 20500 | 11000 |
| 3.4 | 22000 | 11800 |
| 3.6 | 24000 | 13000 |
| 3.8 | 26000 | 15000 |
| 4 | 29000 | 18000 |
| 4.2 | 32000 | 22000 |
| 4.4 | 36000 | 27000 |
| 4.6 | 42000 | 34000 |
| 4.8 | 49000 | 43000 |
| 5 | 58000 | 52000 |

| Values in clear area from BS 1881 | Values in shaded area assumed, to be used for comparison purposes only |
|---|---|



# Appendix E

# Publications based on this thesis

# Appendix E Contents

Hughes, P. and Glendinning S. (2004). Deep dry mix ground improvement of a soft peaty clay using blast furnace slag and red gypsum, Vol 37, pp 205-216.

Hughes, P and Glendinnig S. (2005). Development of a Soil Mixing Binder Using Waste Materials, 1st International Symposium on Waste Engineering (WasteEng 2005), Albi, France

# Deep dry mix ground improvement of a soft peaty clay using blast furnace slag and red gypsum

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## Abstract

The paper describes a field trial using deep *in situ* dry soil mixing to stabilize soft peat deposits beneath the route for the Channel Tunnel Rail Link in the SE of the UK. The aim of this trial was to evaluate whether a ground granulated blast furnace slag (GGBFS)–red gypsum mixture could be used as a replacement for a cement-based binder. Red gypsum is produced as a co-product to the production of the white pigment titanium dioxide. Trial columns were installed adjacent to the main stabilization works. A combination of *in situ* and laboratory testing has shown that the blended binder was as effective as ordinary Portland cement in increasing the strength of the peat. The blended binder columns also displayed similar durability characteristics. Using the alternative binder required no alteration to existing equipment and caused no reduction in production rates. The minerals formed in the samples from the GGBFS–red gypsum columns were of the type formed by pozzolanic reactions and the strength of the GGBFS–red gypsum columns was dependent on achieving a pH of 10.5. The paper recommends that peat samples must be stored in airtight containers prior to initial design mix testing. If required, the pH of the soil–binder mixes should be adjusted (using a small addition of lime) during the treatment process.

*Keywords: case studies, geomaterials, in situ tests, waste management*

Deep *in situ* soil improvement using the dry mix technique has been pioneered in Scandinavia and Japan where very soft soils of high water content are prevalent (Ahnberg *et al.* 1995, 2001; Okumura 1997). The process may be achieved by a variety of methods. One example involves rotating a mixing tool into the ground to the required depth of treatment. Once this has been achieved the rotation is reversed and the tool is withdrawn while binder is pumped by compressed air through apertures in the tool, mixing binder with the soil. Because of the orientation of the fins, this process achieves a degree of compaction through the length of the column. The process is illustrated in Figure 1.

The technique has not been used in the UK until recently, primarily because of the prevalence of stiff overconsolidated soils and the lack of UK-based contractors with experience of the dry soil mix process. However, the deep dry mixing technique was used in

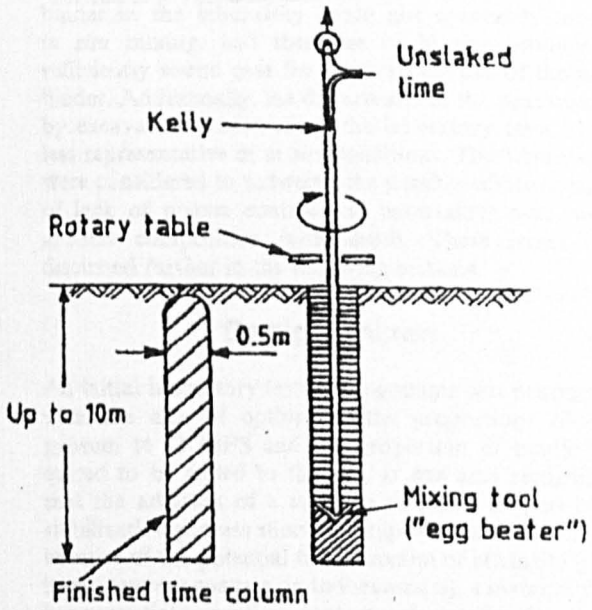


Fig. 1. An illustration of the 'dry mix' process (after Broms & Bomen 1979).

2001 to treat approximately 5 m of soft clay and peat during the Channel Tunnel Rail Link Contract 440 (CTRL 440) located near the village of Sandling in Kent, SE England. The contract involved treating an area of 4250 $m^2$ using columns with a 91% area coverage. The RLE contract specification required the columns of treated ground to achieve a minimum undrained shear strength of 100 kPa and a minimum Young's modulus of 10 MPa (Hansson *et al.* 2001), within 28 days.

The majority of the cost for this type of stabilization is considered to be the price of the binder used. The treatment described above required 3000 tonnes of cement at £25 per tonne to treat 21 000 $m^3$ of soil with a binder concentration of 200 kg $m^{-3}$ soil. Therefore recent research has concentrated on reducing the cost of the binders through experimenting with the whole or partial substitution of waste or marginal materials for cement. Potential replacements have include pulverized fuel ash (PFA), lime and ground granulated blast furnace slag (GGBFS).

Beretka *et al.* (1996) have identified the potential for the use of gypsum-based industrial by-products as alternative sources of cement. The use of these materials has two potential cost advantages: as wastes they have little



1470-9236/04 $15.00 

Table 1 *Composition of red gypsum (after Peacock & Rimmer 2000).*

| Component | Content (by dry weight) |
|---|---|
| | (%) |
| $CaSO_4.H_2O$ | 58.5–59.3 |
| $Fe_2O_3.H_2O$ | 32.9–36.6 |
| Ti | 1.0–1.3 |
| Al | 0.1–0.8 |
| Mg | 0.5–0.6 |
| Mn | 0.2–0.5 |
| Si | 0.9–0.5 |
| Cl | 0.002–0.2 |
| | (mg $kg^{-1}$) |
| Cr | 500–800 |
| Zn | 200–400 |
| Sr | 100–300 |
| Ni | 50–60 |
| Co | 20–30 |
| Ba | 1–3 |
| Pb | 1–2 |

or no associated production cost; and re-use of such a material would negate the need for expensive disposal. Huntsman Tioxide produce a co-product gypsum whilst manufacturing titanium oxide powder, a commonly used white pigment. Globally, Huntsman produce 925 000 tonnes per year of 'red gypsum' (red because of iron content) as a filter cake during the neutralization of sulphuric acids at the end of the titanium oxide production process. A large volume of white gypsum is also produced but this can be used in the production of plasterboard. The composition of red gypsum is detailed in Table 1. Despite the presence of various metals red gypsum has been successfully used to improve agricultural soils, but this application uses only a small proportion of the material produced. Because of its previous use in soil conditioning, comprehensive leachate testing data were available for red gypsum. These data could be made available to any other users of this co-product. Because of this, red gypsum was selected for experimentation into its potential as a partial replacement for cement in soil stabilization.

Peat is known to be problematic for cement-based stabilization techniques as the setting process of cement is retarded by the interaction of the calcium ions and organic matter. Studies have shown that GGBFS can be effective in the stabilization of organic soils (Nenad 1999). Indeed, Hebib & Farrel (2003) have identified its use in combination with gypsum as a potential stabilizer for peat-based soils. For these reasons it was chosen to investigate a binder that combined the co-product red gypsum with GGBFS.

The aim of this field trial was to investigate the potential of using a GGBFS–red gypsum binder for the improvement of very soft peat *in situ* and to compare its performance with that of ordinary Portland cement (OPC). A trial using these alternative binders was conducted adjacent to the ground works for the Channel Tunnel Rail Link. This meant that equipment and material were readily available and a direct comparison of the efficiency of the installation process using the alternative binders with that using OPC could be made. A further advantage was that although laboratory trials could be conducted to assess the most promising binder mixes, the techniques used to mix the peat with the binder in the laboratory could not accurately model *in situ* mixing, and therefore could not produce a sufficiently sound case for the practical use of the new binder. Additionally, the disturbance to the peat caused by excavation would cause the laboratory tests to be less representative of *in situ* conditions. The advantages were considered to outweigh the possible disadvantages of lack of precise control and uncertainty over exact ground composition with depth. These issues are discussed further in the following sections.

## Design mixes

An initial laboratory testing programme was conducted with the aim of optimizing the proportions of red gypsum to GGBFS and the proportion of binder required to be added to the soil. It was also recognized that the addition of a sulphate to a soil as part of a stabilization process should be approached with caution because of the potential for formation of ettringite (and its subsequent conversion to thaumasite), a material that has potential to swell on contact with water and severely weaken the stabilized material (Mitchell & Dermatas 1990; Snedker 1996; BRE 2001). The potential for ettringite–thaumasite formation was also investigated.

### Strength

GGBFS and red gypsum were mixed in varying proportions by mass. Water was then added to achieve a water content similar to that produced when 200 kg $m^{-3}$ of dry binder is mixed with the peat at its natural water content, and tested for strength in undrained triaxial compression. The results, although not conclusive, indicated that the 75:25 GGBFS–red gypsum mixture produced the highest strength. As the most significant cost savings (the maximum use of red gypsum) would be achieved by using the highest possible proportion of red gypsum, a combination of 25:75 GGBFS–red gypsum was also selected for the trial, as it produced only marginally weaker material than the 75:25 mix.

Bulk samples of peat were taken from the site of the proposed field trial and hand mixed in the laboratory with GGBFS–red gypsum binders at their natural moisture content to determine the proportion of binder required to stabilize the soil. The samples were compacted using a tamping bar to a bulk density of 1.1 Mg $m^{-3}$ within a 38 mm diameter mould, transferred to plastic sample tubes, sealed, and allowed to

cure at 20° C for 7, 28 and 56 days. The target bulk density was that of the *in situ* columns and has been found to be an important parameter in laboratory-based design of dry mix columns (Ekman & Holmgren 2002). For comparison, similar samples were prepared using OPC and cured under the same conditions.

Undrained triaxial compression tests, pH tests and X-ray diffraction (XRD) analysis were performed on all the samples. These tests concluded that, although the modified soil had greater shear strength than the original peat, and increasing the amount of binder increased the shear strength of the samples, there was no apparent increase in strength with curing time. This implied that pozzolanic (cementitious) reactions had not taken place. Soaking of the samples for 24 h under water prior to testing was found to reduce the measured strength by *c.* 25%. On average, the samples mixed with cement were 65% stronger and suffered less strength reduction when soaked than the samples mixed with GGBFS–red gypsum, but were weaker than the strengths measured in the *in situ* cement columns in the field.

Investigative testing into the possible cause of this problem found that the pH of the peat samples delivered to the laboratory was below pH 2. This was surprisingly low compared with recorded values of the order of pH 7 for natural peat in the field (Ekman & Holmgren 2001). The addition of the GGBFS–red gypsum binder increased the pH of the peat, with larger quantities of binder producing greater increases in pH. However, pH values rarely exceeded pH 8, significantly below the value required for pozzolanic reactions to take place.

## Ettringite and thaumasite formation

To investigate the potential for ettringite–thaumasite formation, a set of samples were cured through a temperature cycle of 20° C–4° C–20° C and subsequently soaked in water. These conditions were considered conducive to ettringite–thaumasite formation, based on the work of Snedker (1996). XRD testing conducted on these samples indicated the presence of the mineral pyrite; however, no evidence of ettringite or thaumasite was found. There was no obvious difference between the mineral composition of untreated samples and the samples treated and cured for different lengths of time, indicating that curing time had no determinable mineralogical effect.

These tests showed that using a binder of GGBFS and red gypsum improved the strength and durability characteristics of the peat. However, the low pH of the peat did not provide the required conditions for pozzolanic (cementitious) reactions to take place and hence no long-term stability was achieved. Therefore, the most probable reason for the low pH values of the peat was oxidation of the pyrite after exposure to the air. Unfortunately, it is not possible to check for the oxidation products of pyrite using XRD as they are amorphous. With no pozzolanic reactions no ettringite or thaumasite could form.

Table 2 *Binder mixes and concentrations.*

| Mix | Concentration (kg m$^{-3}$ soil) | Ratio (by mass) |
|---|---|---|
| A/G75/200 | 200 | 75% red gypsum, 25% GGBFS |
| B/G75/250 | 250 | 75% red gypsum, 25% GGBFS |
| C/G25/200 | 200 | 25% red gypsum, 75% GGBFS |
| D/G25/250 | 250 | 25% red gypsum, 75% GGBFS |
| E/C100/200 | 200 | 100% cement |

Despite the results, it was considered that the GGBFS–red gypsum binder had potential for use *in situ* where the mixing process meant that the soil was not exposed to the air, thus preventing the oxidizing conditions that caused the low pH values. A selection of binder mixes to be used in the field trial (see Table 1) was designed on the basis of the 'binder-only' test results. The proportion of binder used for stabilization using the dry mix process was based on the experience gained during the execution of the CTRL stabilization works. It was noted that the proportions selected were in line with those used by Hebib & Farrel (2003). In addition to the GGBFS–red gypsum mixes a number of dry mixed columns using OPC were installed so that a comparison could be made. The nomenclature in the left-hand column of Table 2 will be used throughout the rest of the paper to describe the different columns in the trial.

# The field trial

The regional geology around the location of the trial site has been described by Ekman & Holmgren (2001) and broadly consisted of soft marine deposited sedimentary rocks of various lithification known as the Lower Greensand Formations and the Gault Clay. Two cable percussion boreholes were excavated in proximity to the trial area along with seven trial pits. These demonstrated that the local ground conditions consisted of sands of the Sandgate and Folkstone beds overlain by peaty clay–clayey peat in turn overlain by a thin layer of topsoil. *In situ* testing showed that the peat–clay had a typical undrained shear strength of 10 kPa but contained two very soft layers at *c.* 2.2 m and 4.2 m, with strengths of below 5 kPa. Peat recovered from the boreholes and tested in the laboratory had a water content of 327%, a loss on ignition of 55%, and a Von Prost (degree of humification) value of 9.

A total of 27 columns were installed using the combination of binder concentrations and binder ratios shown in Table 2. Powdered red gypsum and GGBFS were delivered to the site by tanker and mixed in the required proportions in the binder delivery vessel. The columns were then formed in exactly the same way as the OPC

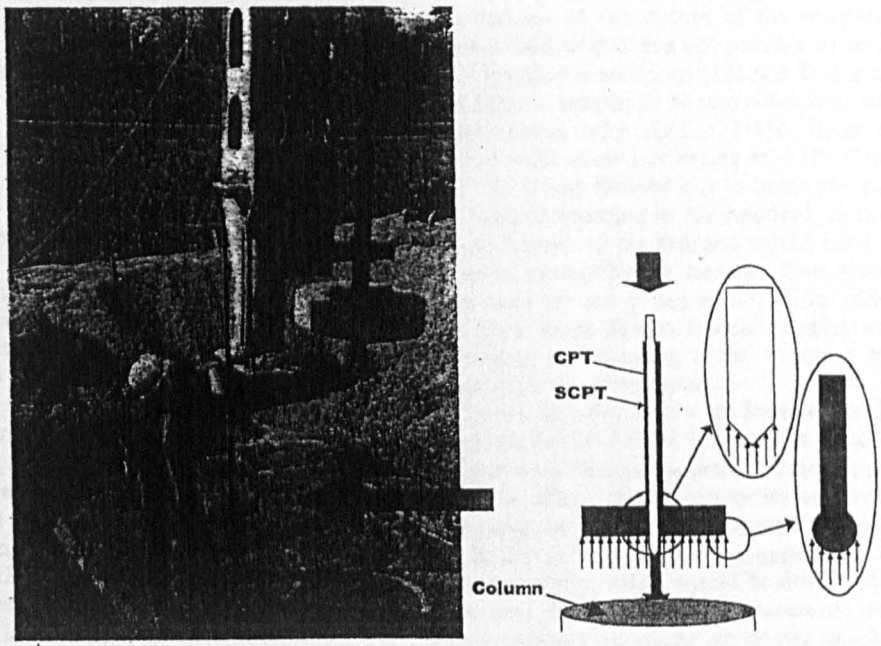


Fig. 2. *In situ* testing equipment.

columns in the full-scale CTRL works, with similar rates of production being achieved. Once the columns were installed there was a delay of 24 h before a 1.5 m layer of fill surcharge was placed over them. This delay was the result of activity on the main site works. Columns on the CTRL route were loaded after a period of 4 h as recommended by Ahnberg *et al.* (2001).

## Testing

A combination of post treatment *in situ* and laboratory testing was used to assess the effectiveness of the ground improvement compared with that of OPC. The most important parameters for comparing the performance with that of OPC were considered to be strength, stiffness, rate of strength development, and durability. Strength and stiffness were assessed *in situ* using the standard column penetration test (SCPT) and in the laboratory using the quick undrained triaxial compression test. The rate of strength development was determined by testing samples after different curing periods. Durability was assessed in the laboratory by soaking samples in water, and by subjecting samples to freeze–thaw, and wet and dry cyclic conditions. The effects of these 'environmental' conditions on the subsequent laboratory strength of the columns was also determined. As previous laboratory testing had indicated that pH was critical for strength development, the pH of all samples was determined. The mineralogical composition of the stabilized soil, including the potential presence of ettringite and/or thaumasite, was investigated using XRD.

## *In situ* testing

Eight columns were tested at 7 and 56 days after installation using the SCPT in which a special two-finned probe was fitted to the end of a standard cone penetration test (CPT) probe (see Fig. 2). The probe was then pushed down through the centre of the column at a constant rate of 20 mm $s^{-1}$ using the equipment used to form the columns. The resistance on the probe was recorded continuously and the undrained shear strength ($C_u$) correlated to 10% of the converted resistance force (Carlsten & Ekstrom 1995).

## Sample recovery

Five rotary boreholes were drilled from the platform layer 1.5 m above the top of the columns using a TR80 rotary rig with a triple tube core barrel and air mist drilling fluid. Cores of 100 mm diameter and 1.5 m long were taken from the top of the columns to a depth of 6 m. Recovery rates were relatively low: columns C/G25/200, D/G25/250 and E/C100/200 had core recovery rates of 80–88%; columns A/G75/200 and B/G75/250 had rates of 70 and 62%, respectively. Cores were stored in sealed plastic tubing to protect sample integrity and prevent any potential oxidation. Triaxial sub-samples (100 mm) were taken approximately every 1 m (or the closest 200 mm long piece of intact core). Once tested for undrained shear strength the samples were retained for pH testing. Freeze–thaw, durability and wet–dry tests were performed on additional samples taken from the remaining available intact core.

## Laboratory testing

### Triaxial compression

Although it would have been desirable to conduct consolidated drained tests on the samples recovered, the number of samples and the requirement to have a standard curing time meant that quick undrained triaxial tests were the only option. The tests were performed in accordance with BS 1377 part 7 (BSI 1990) on samples 100 mm in diameter and 200 mm long. This size was chosen as the diameter of the cores was *c.* 100 mm. Reducing the diameter of the core could have caused further damage to its structure, thus influencing measured strengths and strains. Also, using larger samples was considered to be more representative of the bulk soil mass and, at least in part, to overcome any inconsistencies derived from non-uniform mixing.

The samples were compressed at a constant rate (in this case 1.5 mm $min^{-1}$) while under a nominal confining pressure of 100 kPa. Although it was recognized that this did not represent the *in situ* stress conditions, it replicated previous laboratory testing procedures. The aim of the testing was to compare the performance of the GGBFS–red gypsum columns with cement columns, not to produce design strengths, so consistency was required.

As the axial load was applied, the sample suffered continuous deformation. Throughout the tests, until the sample failed, readings of stress and strain were made at regular intervals. If the sample continued to deform without failing, then the value of stress at 20% strain was recorded as the failure stress. In some cases the samples did not fail before the maximum load of the proving ring had been reached. These samples were cored down to 38 mm and the tests repeated at a 0.5 mm $s^{-1}$ strain rate.

### pH and mineralogical testing

The pH of the samples was determined by the method outlined in BS 1377 part 3 using a Jenway pH meter (model 3150), giving a direct reading of the pH value of the soil suspension in water. Samples were prepared by mixing a 30 g air-dried sample with 75 ml of distilled water for a period of at least 8 h. Mineralogical testing consisted of XRD, scanning electron microscopy (SEM) and petrographic microscope analysis. XRD was performed on 2 g of air-dried sample, ground to no greater than 10 μm in size and compressed onto a slide for analysis. Petrographic analysis was conducted on standard thin sections and SEM was conducted on polished sections.

### Freeze–thaw and wet–dry testing

The freeze–thaw tests conducted in this study were based on the ASTM procedure D560 (ASTM 1996*b*). However, because of the nature of the samples recovered from the field trial it was not possible to create samples of the specified dimensions (101.6 ± 0.41 mm diameter, 116.43 mm in length) so 50 mm cubes were used instead. In accordance with ASTM D560, these cubes were exposed to 24 cycles of freezing at −10° C and thawing at 21° C. It was decided not to brush the samples after each cycle as specified in the standard, as the small and angular nature of the samples would have made them far more susceptible to damage than samples of the dimensions set out in the standard. In addition to the cube tests, seven 38 mm triaxial samples were exposed to freezing and thawing cycles to assess the effect of freeze–thaw on shear strength.

The wet and dry tests were loosely based on ASTM procedure D559 (ASTM 1996*a*). The dimensions of the samples were 38 mm diameter, 78 mm length, again to allow the effects of wet–dry cycles on undrained shear strength to be determined. The drying cycle was carried out at 25° C because the red gypsum used contained structural water, which would be driven off at temperatures over 60° C. As a temperature of over 70° C is highly unlikely to occur at depths much below the ground surface this was considered to be a well-founded amendment to the standard procedure. Samples were tested in undrained triaxial compression after four wetting and drying cycles.

## *In situ* testing results

The response of the GGBFS–red gypsum columns to the *in situ* testing was typified by a peak in shear strength at *c.* 0.4 m depth, dropping to a minimum value at around 1.9 m depth and steadily increasing again to between 4.0 and 4.5 m depth, where the tests terminated (see Fig. 3).

The pure cement columns exhibited higher shear strengths than the GGBFS–red gypsum columns. Generally the shear strength was higher in the columns with a higher concentration of binder for both GGBFS and cement columns. However, in the case of the GGBFS–red gypsum columns, it was difficult to establish which binder ratio was more effective, with no mix showing a consistently higher strength for the entire length of the column. The considerable variation in shear strength was probably attributable to varying initial ground conditions and, possibly, mixing efficiency. As stated above, the boreholes encountered softer ground in parts of the peat layer, and these weaker zones were likely to occur at differing depths spatially across the trial area or may have been absent in some parts altogether.

The fact that it was not possible to perform SCPT tests in the GGBFS–red gypsum columns after 56 days shows that the shear strength continued to increase in the period between 7 and 56 days, which is consistent with the development of pozzolanic reactions.

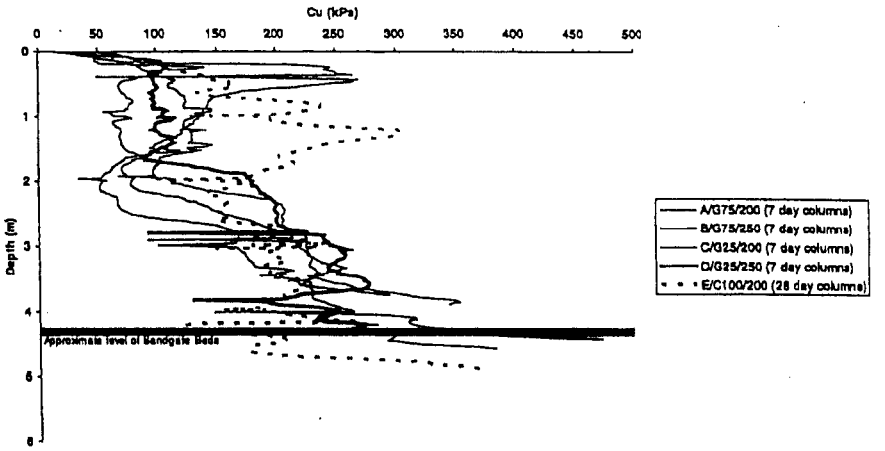


Fig. 3. Undrained shear strength of each column type with depth after 7 days curing (derived from *in situ* testing results).

The GGBFS–red gypsum columns showed considerably greater shear strengths than the samples tested in the initial laboratory testing, thus confirming that the lack of significant improvement in shear strength was probably caused by samples oxidizing before testing; this reduced the pH and thus prevented pozzolanic reactions from taking place.

The GGBFS–red gypsum columns showed a much wider variation in shear strength than the pure cement columns, with shear strengths dropping to 50 kPa in some places. However, as previously stated, this effect may be due to varying initial ground conditions. The trial columns were installed in a different area of the site from the main works, where ground conditions would have been different. This, coupled with the fact that testing was conducted after a longer curing period (56 days as opposed to 21 and 30 days) from the time of the installation, makes comparison difficult.

## Laboratory testing results

### Strength

It can be seen from Figure 4 that mixes A/G75/200 and B/G75/250 exhibited significantly lower shear strengths than the other samples tested, with average undrained shear strengths of 104 and 67 kPa, and minimum and maximum strengths of 35 and 230 kPa and 23 and 94 kPa, respectively. Mixes C/G25/200 and E/C100/200 showed considerable variation, but had approximately the same average shear strengths of 670 and 923 kPa. Mix D/G25/250 showed the highest shear strength, with an average strength of 1946 kPa, and minimum and maximum strengths of 1511 and 3114 kPa, respectively.

All mixes exhibited lower shear strength values between 0.9 and 2.1 m, corresponding to results from *in situ* testing and attributed to natural zones of weakness within the peat layer.

Figure 5 shows the stress–strain results from column D/G25/250; as noted previously the lowest shear strength is exhibited by the sample taken from 1 m depth. It should also be noted that peak strengths were reached at strains typically lower than 1%. However, once the peak strains were reached the post-peak strengths remained relatively high, considerably above the 100 kPa CTRL specification.

Figure 6 shows stress–strain plots from all columns on samples taken from 3 m depth. It can be seen clearly that the samples with greater strengths and stiffnesses reached peak strengths at much lower strains than the weaker samples. Despite this the two stronger samples (C and D) maintained a strength of over 600 kPa past 5% strain.

### Stiffness and strain to failure

Table 3 shows that mixes A/G75/200 and B/G75/250 also exhibited significantly lower Young's moduli, averaging 8.8 MPa and 3.7 MPa, respectively. The other mixes exhibited values between 70 and 230 MPa, with mix D/G25/250 showing the highest value. It should be noted that there was considerable variation in the values of Young's modulus for each mix.

Mixes A/G75/200 and B/G75/250 had the highest average strains at failure, with averages of 10.5% and 7.5%, respectively. Mixes C/G25/200 and E/C100/200 averaged 6.0 and 4.0%, respectively, whereas the average failure strain for mix D/G25/250 was 1.0%.

### Strength development

It can be seen in Figure 7 that the laboratory shear strength of mixes C/G25/200, D/G25/250 and E/C100/200 are higher than those measured during *in situ* testing (increases of 360%, 1240% and 400%, respectively). It would appear, therefore, that strength continued to

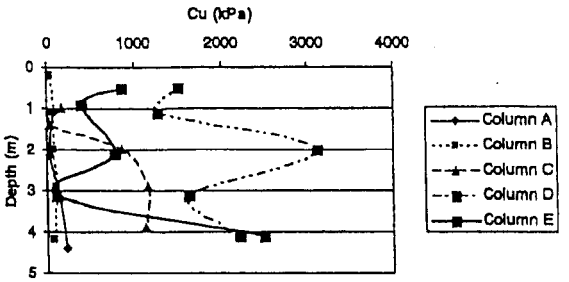


Fig. 4. Results of laboratory strength testing plotted against depth.

develop in the period between SCPT testing and recovery of the samples. In the other columns the shear strength remained similar, with the exception of B/G75/250 where the average undrained shear strength actually reduced by 50%, although this could be attributed to damage inflicted during sample recovery or transport.

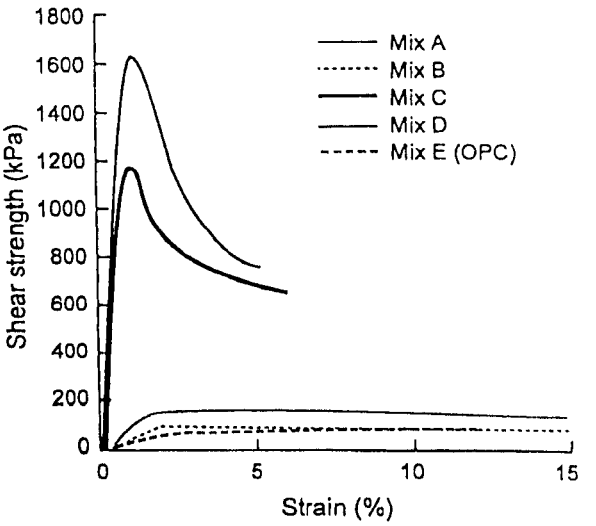


Fig. 6. Stress–strain behaviour of each column mix.

## Durability testing

### Freeze–thaw and wet–dry testing

As can be seen from Figure 8 and Table 4 there was only a limited difference in the shear strength between the control samples (those samples not subjected to freeze–thaw or wet–dry) from C/G25/200 and D/G25/250 and the samples exposed to cycles of freezing and thawing, and wetting and drying. Differences as small as these could have been caused by variations in the quality and consistency of the initial samples. Triaxial testing has shown that there was considerable variation in the strengths of all samples. Figure 9 shows the peak undrained shear strengths from 38 mm quick undrained triaxial samples when soaked and unsoaked. Two of the four samples tested (D/G25/250 at 0.4–0.6 m and E/C100/200 at 4.0–4.2 m) show little change in strength as a result of soaking. However, samples D/G25/250 at 3.0–3.2 m and D/G25/250 at 4.0–4.2 m increased in shear strength when soaked, in the case of the latter by around 100%, but this again could be explained by natural sample variation.

Because of the low strengths of samples from A/G75/200 and B/G75/250 it was not possible to make cube samples from the core so it was only possible to test mixes C/G25/200, D/G25/250 and E/C100/200. Two samples were taken from each mix. Where possible one was taken from as close to 2 m depth as possible in an attempt to test samples from the weaker layer noted in the borehole logs.

All samples remained intact for the first 10 freeze–thaw cycles. Over the course of the first 10 cycles of freezing and thawing samples C/G25/200 at 2.0 m, C/G25/200 at 3.0 m, D/G25/250 at 3.3 m and E/C100/200 at 2.0 m lost little (less than 4%) of their mass,

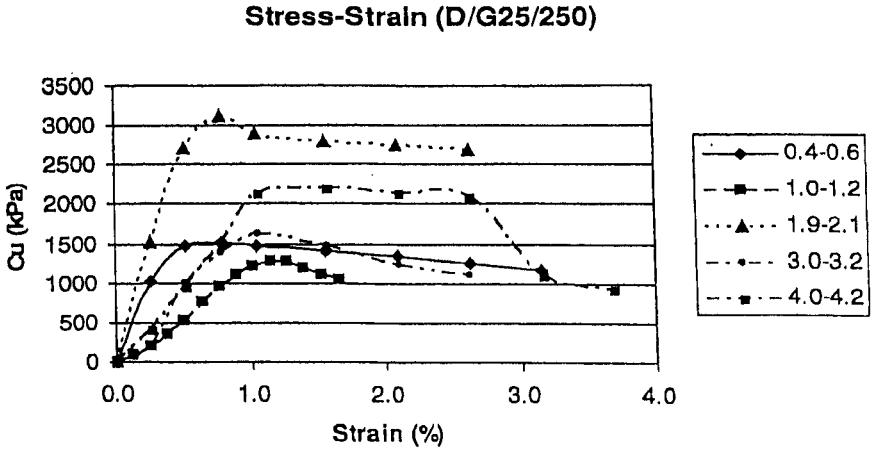


Fig. 5. Stress–strain characteristics for column D.

Table 3 *Summary of strengths and stiffness from laboratory tests.*

| Column | Average undrained shear strength (kPa) | Average Young's modulus (MPa) | $E/C_u$ |
|---|---|---|---|
| A/G75/200 | 81 | 9 | 111 |
| B/G75/250 | 42 | 4 | 95 |
| C/G25/200 | 586 | 123 | 210 |
| D/G25/250 | 1166 | 228 | 196 |
| E/C100/200 | 357 | 71 | 199 |

Table 4 *Results of freeze–thaw and wet–dry triaxial test.*

| Sample | Undrained shear strength (kPa) C/G25/200 | D/G25/250 |
|---|---|---|
| Control | 1341 | 2198 |
| Soaked | 968 | Not tested |
| 4 cycles freeze–thaw | 1198 | 1371 |
| 8 cycles freeze–thaw | Not tested | 1821 |
| 4 cycles wet–dry | Not tested | 1633 |

indicating that they were durable under these conditions, as shown in Table 5. Sample E/C100/200 at 3.0 m began to lose mass after the first cycle and continued to degrade until it had lost more than 30% of its mass by the 10th cycle. Sample D/G25/250 at 1.8 m gained mass steadily (attributed to it taking on more water) until cycle 8, at which point its mass had increased to 1.8% more than its initial value (actually 5% when taken after the end of the freeze cycle). The sample then split into three parts and was effectively destroyed. In the majority of cases the samples proved to be durable against the action of freeze–thaw; the samples of mix D/G25/200 in particular showed little effect. All samples that remained intact after the 10th cycle were tested to the full 24 cycles. For sample E/C100/200 at 2.0 m, between 10 and 15 cycles the mass of the remaining sample did not change significantly but on the 16th cycle it disintegrated. During the remainder of the test the mass of sample D/G25/250 at 3.3 m remained constant whereas samples C/G25/200 at 2.0 m and 3.0 m suffered significant reduction in mass although they did not totally disintegrate.

During the freeze–thaw cycles conducted on the cube samples the dimensions of the cubes were measured at regular intervals. Thus it was possible to gain an indication of the shrink and swell of the modified soil. It was found that the maximum swell occurred in sample E/C100/200 at 3.0 m (0.9%); samples D/G25/250 at 1.8 m and 3.3 m exhibited swells of 0.7 and 0.5%, respectively, but both samples from mix C/G25/200 had maximum swells of 0.3%.

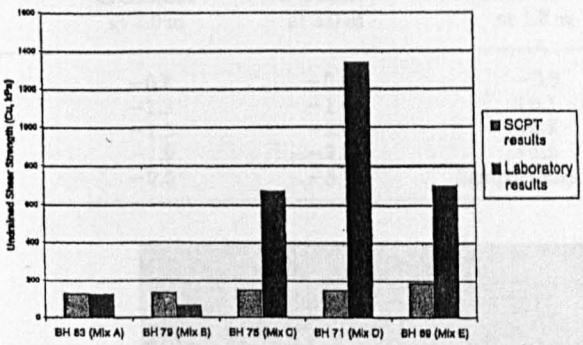


Fig. 7. Comparison of laboratory and *in situ* strength results.

## Mineralogical testing

All samples had pH values between 8.5 and 12.1. Samples from mix A/G75/200 showed the lowest values of pH at 8.5. In no case did the pH values fall to those of the original laboratory samples.

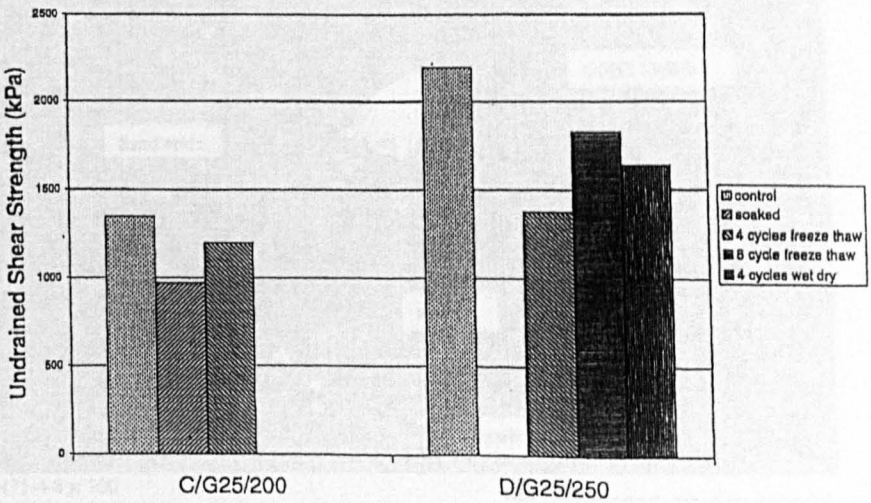


Fig. 8. Undrained shear strength of columns C and D after subjection to freeze–thaw and/or wet–dry cycles.

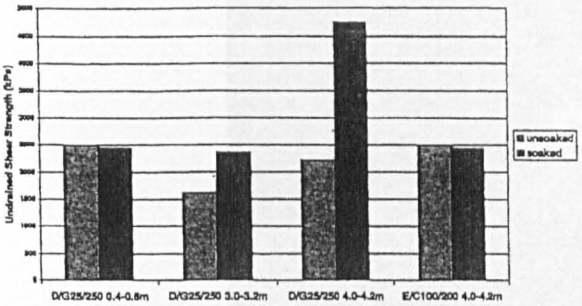


Fig. 9. Undrained shear strength of column D and E after subjection to soaking.

None of the 27 samples tested by XRD exhibited any evidence of the presence of ettringite or thaumasite. There was also no evidence of the presence of red gypsum. Unfortunately, because of their amorphous nature, XRD is not suited to investigating the presence of hydrated cement products.

Petrographic analysis showed that sections taken from the weaker mixes, A and B, contained visibly more organic material than those taken from mixes D, C and E. Also noted was the presence of numerous sand grains. Again, no evidence of ettringite or thaumasite was found.

Polished sections were made from samples taken only from columns D and E. The slides were examined by SEM (see Figs. 10 and 11). Examination showed that the specimens contained very few void spaces, implying that those initially present had been filled with hydrated cement products. Only small areas of organic material were seen to be present, and the presence of some sand grains was noted. Elemental analysis was conducted on several grains observed on the slides, the results of which confirmed the presence of silica grains, particles of unhydrated GGBFS and red gypsum (in mix D), particles of unhydrated OPC (in mix E), and amorphous hydrated cement products in the sample matrix. Again,

Table 5 *Results of freeze–thaw durability tests (first 10 cycles, mass measured at end of thaw).*

| Cycles | Loss of mass (%) | | | | | |
|---|---|---|---|---|---|---|
| | C/G25/200 at 2.0 m | C/G25/200 at 3.0 m | D/G25/250 at 1.8 m | D/G25/250 at 3.3 m | E/C100/200 at 2.0 m | E/C100/200 at 3.0 m |
| 2 | −0.8 | −0.8 | −0.5 | −0.6 | −0.8 | −3.4 |
| 4 | −1.3 | −1.6 | +0.1 | −0.6 | −1.0 | −4.8 |
| 6 | −1.5 | −2.4 | +0.9 | −0.6 | −0.5 | −9.1 |
| 8 | −1.9 | −3.3 | +1.8 | −0.5 | −0.6 | −21.4 |
| 10 | −2.0 | −3.5 | Sample destroyed | −0.5 | −0.5 | −31.9 |

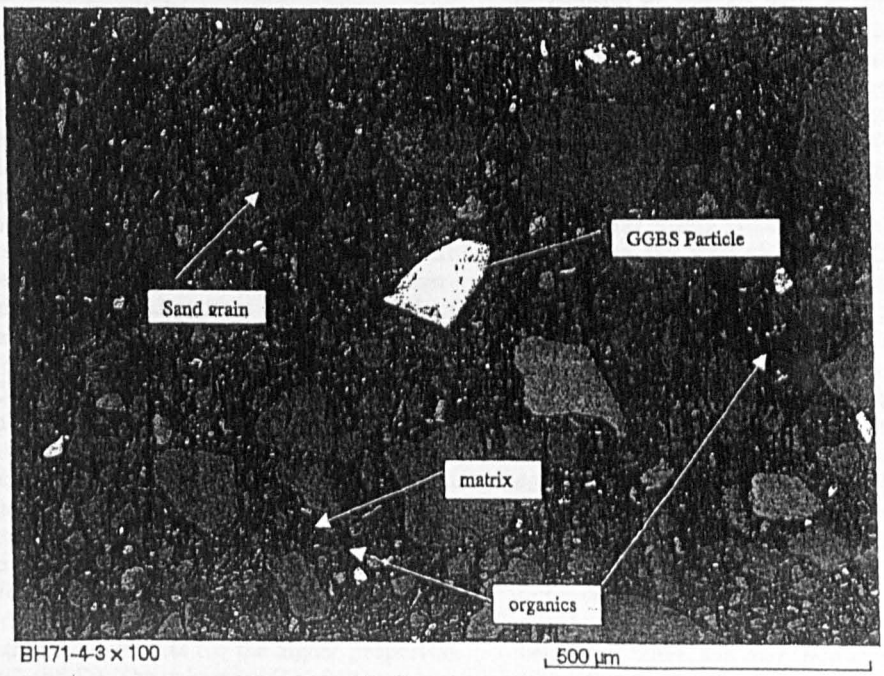


Fig. 10. Polished section from column D.

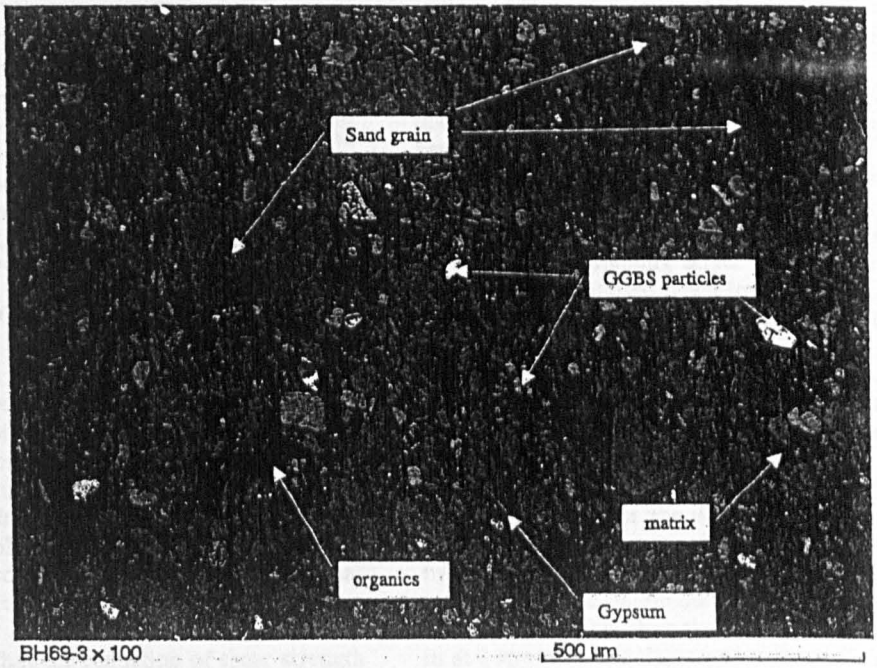


Fig. 11. Polished section from column E.

no evidence was found for the formation of ettringite or thaumasite. Elemental analysis showed the needle-shaped crystals present (see Figs. 10 and 11) to be red gypsum. This indicates that the XRD equipment was not sufficiently sensitive to pick up the red gypsum in the bulk material.

## Discussion

The average strengths measured *in situ* after 7 days of formation were of similar magnitude for all the columns tested, particularly when viewed in the context of the variability of the original ground conditions.

The laboratory testing indicated that C/G25/200 reached the same average strength as that of the cement column mixed at the same binder–soil proportion. However, the same GGBFS–red gypsum binder achieved an average strength of 200% of the OPC column when the binder proportion was increased to 250 kg $m^{-3}$. D/G25/250 and C/G25/200 both exhibited higher Young's moduli than the OPC column, in the case of D/G25/250 nearly three times higher. The strain to failure of D/G25/250 was, however, only 25% of that of the cement column, whereas the lower proportion of binder produced an increase to a strain at failure of 33%. Looking at the ratio of Young's modulus to undrained shear strength ($E/C_u$), in Table 2, the values for two columns containing the higher proportion of gypsum (A and B) were approximately half that for the higher proportion of gypsum (C and D). The values for C and D were very similar to that of the pure cement samples. Despite the relatively low strains to failure exhibited by the stronger samples, post-peak strengths remain higher than the 100 kPa specified for the CTRL project, in most cases several times higher.

Previous workers (Hebib & Farrel 2003) have noted an increase in stiffness with an increase in confining pressure. Analysis of the results from the GGBFS–red gypsum improved columns did not show such a relationship. This can be explained, however, as the variability in the initial ground conditions having much greater control over the final properties of the stabilized peat, and initially weaker zones were found to correspond to increasing depth.

There was strong evidence that pH was a controlling factor over the strength of the GGBFS–red gypsum columns. Overall the results from the pH testing did not indicate that samples with higher pH values produced higher shear strengths, but that high shear strengths were not reached unless the pH was above 10.5–11. This is illustrated in Figure 12. This should not be considered surprising as a pH above 10.5 is required to bring alumina and silica into solution to produce cementitious compounds (Eades & Grim 1966) and thus produce appropriate conditions for pozzolanic reactions to occur. Kuno *et al.* (1989) also noted that increased levels of humic acid in organic soils influenced the soil improvement factor. Mixes with the higher proportion of red gypsum A/G75/200 and B/G75/250 had pH values below this range, and they were also the weakest samples to be tested. As discovered with the initial laboratory

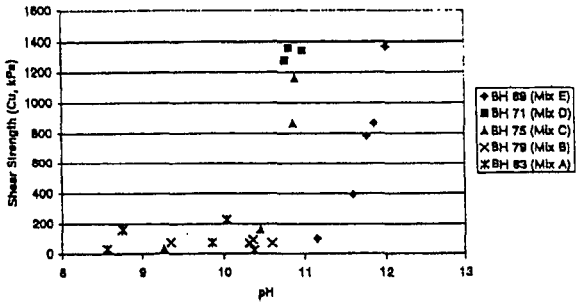


Fig. 12. Comparison of undrained shear strength and pH of sample.

testing, if there is a possibility for oxidizing reactions to reduce the pH of soil samples, these samples must be stored in airtight containers prior to testing. This is particularly important in the process of laboratory-based design of *in situ* mixing of cementitious binders. The extremely high pH of cement means that this is less important for OPC columns, although it is possible that it may have an effect on the calculated binder concentrations. The effect of pH on the initial laboratory samples has meant that the reduction of shear strength values (as recomended by Hebib & Farrel (2003) and EuroSoilStab (2001)) of laboratory results was inappropriate in this case. Although it is recognized that strength reduction factors are important in most cases, as they take into account, for example, mix energy and efficiency between the laboratory and the field, it is important that they are not applied without due regard for any other difference between laboratory and field conditions that may be in operation.

Soaking did not have a significantly detrimental effect on the undrained shear strength of column D/G25/250 at any depth tested and it compares well with the cement column. The cause of the increase in strength at 4.0–4.2 m depth has not yet been identified, but is accompanied by an increase in mass (observed in the cube test). As the cube sample increased steadily in mass and then suddenly split into three pieces, the possibility of ettringite formation was considered, the needle-like crystals at first increasing the strength, with subsequent swell ultimately reducing strength. However, neither visual observation of the failed sample nor XRD results supported this hypothesis.

It would seem that freezing and thawing–wetting and drying do not have a significant impact on the undrained shear strength of the treated peat, but it was not possible to make a comparison with the performance of the OPC columns because of lack of intact samples available for testing.

The mineralogical testing results strongly indicate that pozzolanic reactions have taken place in the GGBFS–red gypsum and cement mixes and also that ettringite and thaumasite were not present in the samples tested. Hebib & Farrel (2003) noted in their conclusions on testing performed on cementitiously stabilized peat that cement particles fill the large void spaces that are characteristic of such soils, but that no interaction between hydrated cement products and the organic material could be observed. The samples analysed could best be described as a matrix of GGBFS, red gypsum, sand, hydrated cement products and organic material, and it was probable that the organic material present had not interacted with the cement. It was noticeable that samples containing larger percentages of organic material also exhibited much lower shear strengths, the strength of the matrix clearly being reduced by increased amounts of organic material.

The SEM analysis conducted on the field trial samples revealed the initial soil to be a mixture of clay and sand with variable but relatively small quantities of organic material and no voids. Therefore the soil could have better been described as a peaty clay than peat or clayey peat. The reason for the discrepancy between this and the available site investigation data could be explained by the fact that boreholes and trial pits were positioned by contractors to obtain best quality information for the CTRL route rather than the field trial. Lateral variation in soil strata to this degree, over these distances, is not uncommon. It is an unfortunate fact that in projects of this type, where field trials are appended onto large commercial projects, the practicalities of the commercial project must take priority.

## Conclusions

From the process of conducting the field trial and subsequent *in situ* and laboratory investigations described above, the following conclusions were drawn.

A GGBFS–red gypsum binder can be substituted for OPC in the dry mix process without any modifications to existing plant and equipment or a reduction in the efficiency of the installation process. No extra operator training or personal protective equipment is required.

From considerations of strength and durability and economics, the most effective binder was considered to be mix C (25% red gypsum, added at 200 kg $m^{-3}$).

The average 7 day strengths of GGBFS–red gypsum columns were similar to those achieved in the OPC columns. Longer-term strengths were found to be higher than those of the OPC columns when the proportion of binder was increased from 200 to 250 kg $m^{-3}$. The increase in the proportion of the binder reduced the strain to failure, but post-peak strengths remained in excess of the design specification. Hence the alternative binder was at least as effective as OPC as a stabilizing agent for peaty clay.

From the tests performed during this study, the strength of GGBFS–red gypsum columns tested did not appear to be significantly detrimentally affected by soaking, freeze–thaw or wet–dry cycles, so it may be argued that the columns are reasonably durable. Again, the

alternative binder produced columns that were at least as durable as the cement columns under the same conditions. However, only a limited number of samples could be tested and further testing of the long-term strength characteristics is recommended.

No evidence of ettringite or thaumasite has been found in any of the samples tested, indicating that, in the short term at least, the GGBFS columns are not susceptible to sulphate attack.

Mineralogical investigations have shown that the minerals formed in the samples from the GGBFS–red gypsum columns were of the type formed by pozzolanic reactions. It can therefore be reasonably concluded that pozzolanic reactions between the GGBFS and the red gypsum were taking place. This was further evidenced by the observed pH dependence of the strength of the GGBFS–red gypsum columns.

It is recommended that, if there is a possibility for oxidizing reactions to reduce the pH of soil samples, these samples must be stored in airtight containers prior to initial design mix testing. Furthermore, if there is any doubt over the pH of the soil–binder mixes this should be adjusted (using a small addition of lime) during the treatment process.

**Acknowledgements.** The authors would like to thank the EPSRC and Huntsman Tioxide Ltd for sponsoring the research project of which this trial is a part. In addition, the authors would like to thank the EPSRC for funding XRD apparatus at Newcastle University (Grant GR/R62113/01), the use of which was invaluable to this research. Also acknowledged is the input from the Stent–Hercules partnership, which undertook the field trials and *in situ* testing, and provided advice on design and laboratory testing. Thanks are extended to CTRL for granting permission for the trial to take place and access to the site.

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Received 15 January 2004; accepted 7 July 2004.

# Development of a Soil Mixing Binder Using Waste Materials

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## Abstract

Huntsman Tioxide produce a co-product "Red Gypsum" (red due to iron content) as a filter cake during the neutralisation of sulphuric acids at the end of the Titanium dioxide ($TiO_2$) production process. Globally, Huntsman produce 925 000 tonnes per year of red gypsum. The majority of the material goes to landfill, the rising cost of which has made it essential to find alternative uses.

At present the majority of the cost incurred in soil mixing ground improvement is the cost of the binder, typically Portland cement. In addition to the financial cost there is also the environmental cost of quarrying and processing of materials to produce Portland cements. Gypsum based industrial bi-products have been identified as alternative sources of cement (Beretka et al, 1996). Using these materials has two advantages: they have little or no production cost; and the re-use of such material would negate the need for expensive disposal.

Laboratory trials have been used to investigate the properties of red gypsum on its own and when mixed with Pulverised Fuel Ash, Ground Granulated Blast Furnace Slag, Lime and Steel Slag at a range of water contents. An assessment of samples was made on the basis of Unconfined Compressive Strength at 28 days curing. It was found that a Red Gypsum : Ground Granulated Blast Furnace Slag mix achieved the highest unconfined compressive strength (up to 39 MPa) and was selected for further investigation as a binder.

This binder was mixed with silty sand at percentage additions of 5, 10 and 20% by dry weight. These were tested for un-drained triaxial compressive strength, x-ray diffraction, and scanning electron microscope (SEM) analysis, and compared with Portland cement binder. The red gypsum based binder compared favourably with results using Portland cement, the strength gain being attributed to the formation of typical hydrated cement minerals.

**Keywords:** Soil Mixing, Waste re-cycling, Laboratory Trial, Red Gypsum

## Introduction

Deep in-situ soil improvement using the dry mix method has been pioneered in Scandinavia and Japan where very soft soils of high water content are prevalent (Ahnberg et al, 1995, 2001; Okumura, 1997). One method of achieving the process involves rotating a mixing tool into the ground to the required depth of treatment. Once this has been achieved the rotation is reversed and the tool is withdrawn while binder is pumped by compressed air through apertures in the tool mixing the binder with the soil (see Figure 1). Because of the orientation of the fins, this process achieves a degree of compaction through the length of the column. An alternate method involves spreading the binding agent across the surface of the soil requiring improvement then mixing the binder into the soil using a rotovator. After the binder is sufficiently mixed with the soil the area is compacted, typically using a vibrating roller. This technique only improves the soil to relatively shallow depths but is a useful technique for highway construction.

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Binders typically used in these applications are Portland Cement or Lime. The cost of these materials often makes up the majority of the cost of the ground improvement.

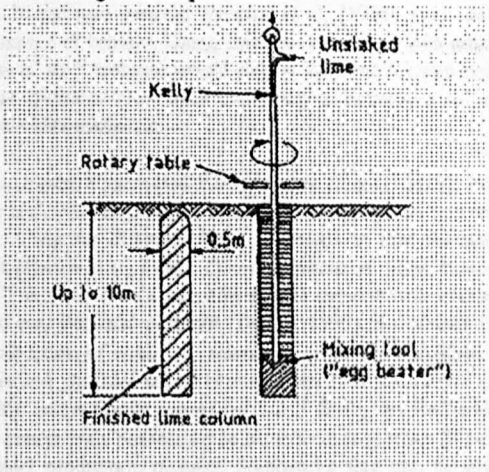



*Figure 1 An illustration of the 'dry-mix' process (after Broms and Bomen, 1979).*

Huntsman Tioxide produce a co-product "Red Gypsum" (red due to the presence of iron oxide) as a filter cake during the neutralisation of sulphuric acids at the end of the Titanium dioxide production process. Titanium dioxide powder is widely used white pigment in items such as paints or plastics. Globally, Huntsman produce 925 000 tonnes per year of Red Gypsum. Some Red Gypsum is sold as an agricultural soil conditioner but this application only uses a small proportion of the material produced. The remainder of the material goes to landfill, the rising cost of which has made it essential to find alternative uses. The composition of Red Gypsum is shown in Table 1.

| Component | Content (by dry weight) |
|---|---|
| | % |
| $CaSo_4.H_2O$ | 58.5-59.3 |
| $Fe_2O_3.H_2O$ | 32.9-36.6 |
| Ti | 1.0-1.3 |
| Al | 0.1-0.8 |
| Mg | 0.5-0.6 |
| Mn | 0.2-0.5 |
| Si | 0.-0.5 |
| Cl | 0.002-0.2 |
| | $mg\ kg^{-1}$ |
| Cr | 500-800 |
| Zn | 200-400 |
| Sr | 100-300 |
| Ni | 50-60 |
| Co | 20-30 |
| Ba | 1-3 |
| Pb | 1-2 |

*Table ., Composition of Red Gypsum (after Peacock and Rimmer, 2000).*

Investigations at Strathclyde University and the University of Newcastle upon Tyne (Grant, 1997; Simpson, 2001; Hughes and Glendinning, 2004) demonstrated that when mixed with other waste materials Red Gypsum could be modified to form a Pozzolanic cement. Beretka et al, (1996) also identified the potential of using gypsum wastes as alternate sources of cement. Using these materials has two advantages: they have little or no production cost; and the re-use of such material would negate the need for expensive disposal. In light of this it was decided to conduct further studies at Newcastle University to develop a soil mixing binder by mixing Red Gypsum with a variety of other waste materials.

Red Gypsum was mixed with Ground Granulated Blast furnace Slag (GGBS), Pulverised Fuel Ash (PFA) and Steel Slag. Blast furnace slags are obtained from the manufacture of pig iron and contain silica, alumina and lime (Neville and Brookes, 1993). They are different from pozzolans in that the nature of the reactions and the reaction products are different (Harris et al, 1995). Ground granulated blast furnace slag (GGBS), which is the type most available in the UK, is classed as a latent hydraulic cement with compositions broadly intermediate between a pozzolanic material and Portland cements (Taylor, 1990). Pulverised Fuel Ash (PFA) is a synthetic pozzolana created by the combustion of coal. UK ashes are generally classified as low lime PFA, consisting mostly of glassy, hollow, spherical particles called cenospheres. It is a siliceous and aluminous material which on its own possesses very little cementitious properties but if finely divided and mixed with water will react chemically with lime to form compounds possessing cementitious properties (Neville and Brookes, 1993). Steel slag is a by-product of the steel production process. Steelworks slag can be divided into two main types in accordance with their method of production: basic oxygen steelmaking slag (BOS); and electric arc furnace slags (EAF). Although several forms of lime exist, generally it is only quicklime (calcium oxide) and hydrated lime (calcium hydroxide) that are used as binders. Quicklime, which exists either in granular or powder form, is produced from heating chalk or limestone, and hydrated lime, which is generally available as a fine, dry powder, is produced as a result of the reaction of quicklime with water (Al-Tabbaa and Perera, 2001).

## Binder Test Programme

| Addition to Red Gypsum | Red Gypsum : Additive mix ratios tested |
|---|---|
| Pulverised Fuel Ash | 50:50, 30:70 |
| Steel Slag | 90:10, 70:30, 50:50, 30:70, 90:10 |
| GGBS | 90:10, 80:20, 70:30, 60:40, 50:50, 40:60, 30:70, 20:80, 90:10 |

*Table 2. Binder combinations tested.*

Table 2 shows the Red Gypsum : waste mix ratios tested. These were initially mixed at a range of water contents in order to assess the optimum water content for developing high strengths. A 1% addition of lime was made to each mix in order to ensure that the pH was over 10.5 to facilitate pozzolanic reactions.

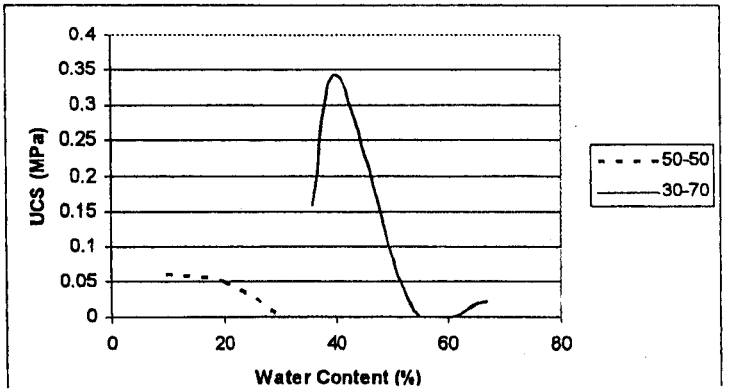
Samples were prepared by compacting material using a 2.5kg rammer into a 1 litre compaction mould as per BS 1377, part 4, clause 3.3. 38mm steel sample tubes were then driven into the centre of the mould to obtain individual samples. As the water content of the samples was increased they became unsuitable to be compacted by the method described above. Where this occurred samples were poured into the sample moulds and vibrated using a vibrating poker to ensure the absence of air pockets. All samples tubes were immediately sealed with wax once binder samples had been taken.

Samples were cured in the steel sample cylinders for 28 days in a temperature controlled store room (20 °C, 55% relative humidity). Once the curing period was complete samples were extruded, trimmed to 76mm in length and tested immediately.

The strength of the samples was assessed by unconfined compressive strength testing conducted according to BS 1377 part 7, clause 7. Water content of all samples was assessed by oven drying after testing. Samples were retained after strength testing for SEM and XRD analysis.

## Binder Results

Unconfined Compressive Strength (UCS) tests conducted on Red Gypsum – PFA binder mixes indicated that of the two ratios tested the 30:70 yielded the highest strength at a water content of 40.5% (Figure 2).



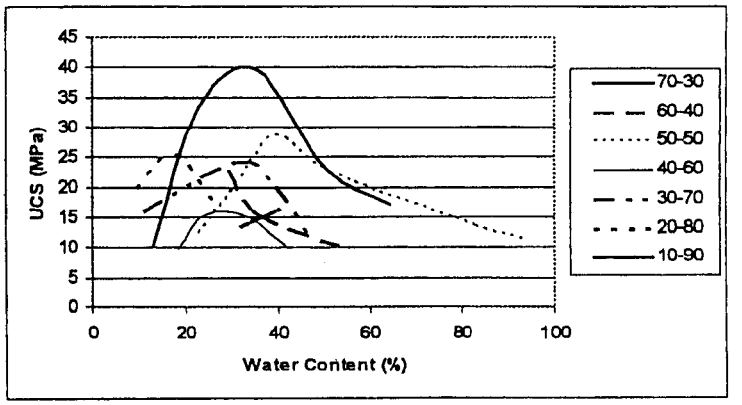



*Figure 4. Red Gypsum – GGBS UCS results.*

All of the Red Gypsum – Steel Slag mixes exhibited the highest strengths (see Figure 2) at the lowest water contents tested. Attempts were made to test mixes at lower water contents. However the samples produced were very brittle and broke apart before they could be tested. The highest strength achieved was 4.51 MPa in the 30-70 mix at a water content of 18.6%.

The highest strength achieved in the Red gypsum – GGBS mixes was 39.7 MPa in the 10:90 ratio at 35.4% water content (see Figure 4). However, the 50:50 mix also produced a significant strength (28.6 MPa at 39.6% water content).

## Binder Mixing Conclusions

Of the three wastes trialled the combination of Red Gypsum with GGBS was clearly the most effective binder. Given that one of the objectives of the research was to develop a binder to make use of large volumes of Red Gypsum and that GGBS already has many applications within the construction industry it was decided to use a 50:50 mix in the soil mixing phase of the research.

## Soil Mixing

Alluvial silty sands are frequently problem soils in the UK due to their relatively weak strength and lack of cohesion. Therefore it was decided to asses the effectiveness of the Red Gypsum – GGBS binder by mixing it with a laboratory manufactured silty sand at a range of percentage additions (5, 10 and 20% by dry weight) and comparing the un-drained shear strengths with samples prepared with a Portland cement binder.

The laboratory mixed soil was composed of 70% medium silica sand and 30% grade E Kaolin. It was mixed with the Red Gypsum – GGBS binder at its optimum water content, (calculated in accordance with 2.5 kg compaction test, BS 1377, part 4, clause 3). After mixing, samples were compacted into 38 mm diameter sample moulds to a bulk density of 2.1 $Mg/m^3$. In order to represent different conditions in the field samples were cured in un-soaked (sealed into the sample tubes using sealing wax) and soaked (tubes left open and submerged in a water filled curing tank and an 18kPa surcharge applied). The un-soaked curing conditions represent shallow mixing above the water table; the soaked curing represents deep mixing below the water table under higher confining pressures.

Samples were tested for undrained triaxial compressive strength in accordance with BS 1377 part 7 clause 8 after curing periods of 7, 14, 28, 56 and 112 days. 3 samples were tested at each curing period.

**Soil Mixing Results**

As can be seen in Figure 5 the addition of Red Gypsum – GGBS binder increased the strength of the silty sand progressively with curing time when cured in un-soaked conditions. Greater additions of binder caused greater increases in strength. For the Red Gypsum – GGBS samples the strength increased most rapidly between 0 and 56 days after which the rate of strength increase dropped off, although strength was still increasing at 112 days. The Portland cement control samples (20% concentration) increased in strength much more rapidly than the Red Gypsum – GGBS samples, more than 50% of the strength increase occurring in the first 7 days. However, by 28 days the Portland cement and Red gypsum shear strengths were approximately the same.

A similar pattern can be seen in the soaked curing samples (Figure 6) but with soaked samples achieving shear strengths approximately half that of the dry samples. The 20% Portland cement control samples were stronger than the Red Gypsum – GGBS samples throughout. Nevertheless, the Red Gypsum – GGBS samples did achieve 80% of the strength of the Portland cement samples.

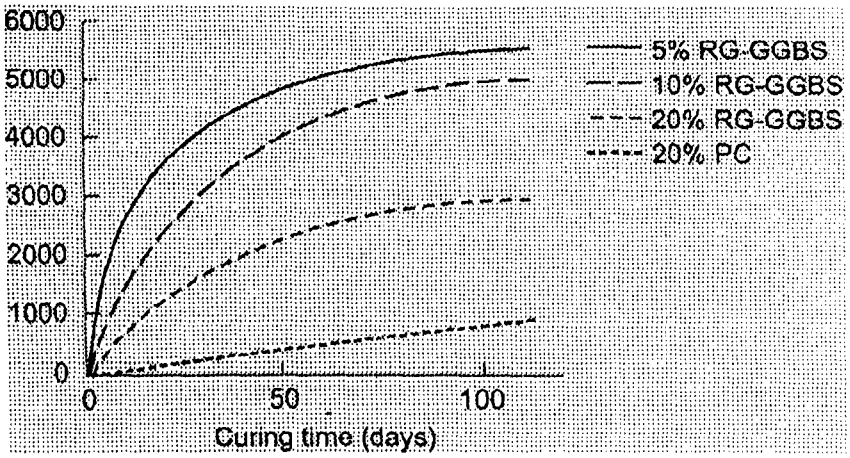



*Figure 5. Un-drained shear strength of un-soaked samples.*

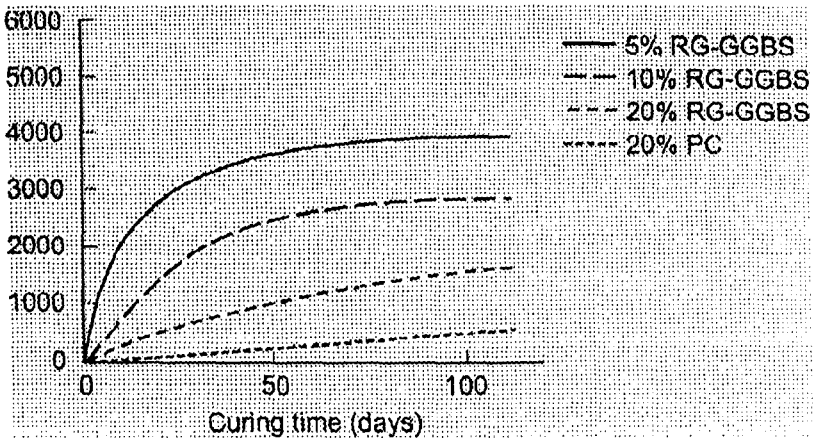



*Figure 6. Un-drained shear strength of soaked samples.*

## Mineralogical Testing

Binder and soil mix samples were retained for X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis. Samples selected for analysis were a sample of 40:60 Red gypsum – GGBS binder, 20% Red Gypsum - GGBS binder mixed with silty sand and 20% Portland cement binder mixed with silty sand (soaked and un-soaked soil mix samples were tested). This analysis was conducted to see which, if any, of the standard hydrated cement minerals formed and also to check for the presence of etteringite and/or thaumasite. These materials that have the potential to swell on contact with water and severely weaken stabilised materials (Mitchell and Dermatas 1990; Snedker 1996; BRE 2001).

The XRD analysis of the binder samples indicated the presence of gypsum and etteringite, but no hydrated cement minerals were evident. However this is probably due to most hydrated cement minerals being X-ray amorphous. Point elemental analysis conducted using the SEM apparatus did however indicate the presence of hydrated cement minerals.

The X-ray diffraction analysis of the soil mix samples indicated that the dry cured Red Gypsum – GGBS sample contained Kaolinite, Gypsum, Etteringite and Quartz whilst the sample cured in soaked conditions additionally contained Portlandite, Calcite and Bassanite.

There was no difference in the results from the Portland cement samples, each contained Portlandite, Calcite, Kaolinite and Quartz. The principal difference in X-ray diffraction results between the Red Gypsum – GGBS samples and the Portland cement controls is the non occurrence of Etteringite and Gypsum in the Portland cement samples.

## Discussion

The initial binder results showed that of the three waste materials mixed with Red Gypsum Ground Granulated Blast Furnace was by far the most effective, although with further development it may be possible to use Red Gypsum – Steel Slag as a binder for applications where only a low strength increase is required. The X-ray amorphous nature of the most common hydrated cement minerals (Calcium Silicate Hydrate, Calcium Alluminate Hydrate etc.) meant that mineralogical analysis of the binder samples could not be conclusive. However, the SEM analysis did indicate strongly that some hydrated cement minerals were present and given the high strengths of the samples it is assumed that the binder did undergo pozzolanic reactions.

Mixing the Red Gypsum – GGBS binder with a laboratory mixed silty sand resulted in the soil increasing in strength significantly, larger quantities of binder yielding larger strength gains. The level of improvement was on 50% lower when the samples were cured in soaked conditions. When compared to an equal percentage addition of Portland Cement binder the Red Gypsum binder achieved only slightly lower strengths in both un-soaked (1% lower) and soaked (16% lower) curing conditions. The Portland cement improved samples increased in strength much more rapidly than the Red Gypsum-GGBS binder, achieving > 50% of the 112 days strength by 7 days. By contrast, the 20% Red Gypsum – GGBS samples took until 14 days in the case of the un-soaked samples and 28 days in the case of the soaked samples to achieve similar improvements. The slower development of strength maybe a disadvantage to the acceptability of the binder for use on site, while many construction specifications require a specified strength to be developed by 28 days (Hanson et al, 2001) more rapid development of strength is frequently desirable.

Of the Red Gypsum – GGBS bound soil samples only the soaked sample exhibited evidence of hydrated cement minerals (Portlandite) although both soaked and un-soaked samples exhibited evidence of the presence of Etteringite. The presence of the Etteringite within the samples is a cause for concern, although during the testing programme no swelling of the samples was observed. Also it should be noted that whilst X-ray diffraction analysis technique used can identify the presence of Etteringite it cannot quantify it and only small amounts may have formed. Differences in the mineral content between the soaked and unsoaked Red Gypsum – GGBS samples are probably due to the higher volume of water available for hydration reactions. Neither of the Portland cement samples exhibited the presence of Etteringite.

## Conclusions

From an engineering standpoint Red Gypsum can be used as an effective soil binding agent in silty sands when mixed with GGBS. Significant improvement of weak soil can be achieved in both soaked and un-soaked conditions suggesting that the binder would be effective in both shallow and deep soil mixing ground improvement. However, slower strength development may limit the use of Red Gypsum – GGBS binders in some applications. Also, the presence of Etteringite in the samples is a cause for caution and extensive durability testing such be conducted before the material is used. Further mineralogical analysis of Red Gypsum –GGBS improved soil involving the testing of larger numbers of samples would also be desirable.

The results compared well with Portland Cement. Provided that costs of producing and transporting the binder can be kept low and that the improved soil can be shown to be durable then Red Gypsum – GGBS can be developed further and employed in civil engineering projects.

### Acknowledgements

The authors would like to thank the EPSRC and Huntsman Tioxide Ltd for sponsoring the research project of which this trial is a part. In addition, the authors would like to thank the EPSRC for funding XRD apparatus at Newcastle University (Grant GR/R62113/01), the use of which was invaluable in this research.

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